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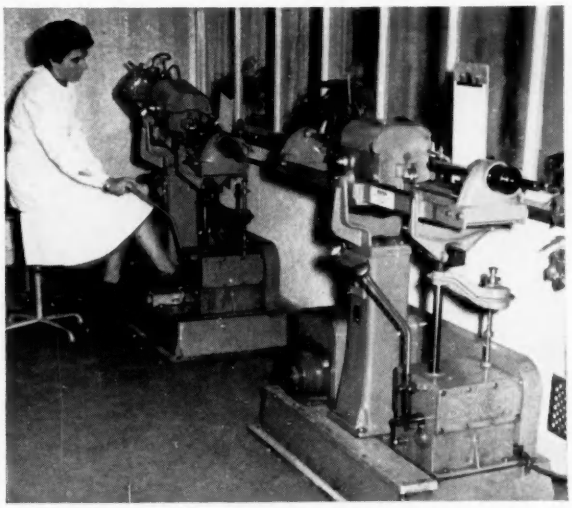

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TEXTILE RESEARCH JOURNAL

VOL. XXVII, NO. 6 JUNE 1957

The Surface of Cotton Fibers

Part I: Native Fibers

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Abstract

The topography of unmodified cotton fibers has been examined microscopically by means of surface replica techniques. The outstanding characteristic of the surface is a system of roughly parallel ridges and grooves spiraling around the fiber at an acute angle (usually 20–30°) to its axis. The pattern of the surface is a reflection of the spiral fibrillar structure of the cellulose beneath the primary wall of the fiber. The average height and distance between ridges is approximately 0.5 micron, and many of the ridges are 10 or more microns in length.

The surfaces of most native cottons are quite similar, although statistically significant differences in the average distance between ridges were found between certain samples. The surface of fibers from unopened bolls is relatively smooth, but assumes the roughness of the typical fiber on drying for the first time. A characteristic surface formation appeared to be associated with fibers known to have undergone compression. Drastic abrasion largely obliterates the normal appearance of the fiber surface.

THE methods of nearly every phase of cotton technology are based on the structural characteristics of the cotton fiber. A special aspect of these characteristics is fiber topography. The response of the surface to physical and chemical environments will often determine the efficiency and utility of the whole fiber in various fields of application. A survey of the microscopic topography of cotton was undertaken at this laboratory in order to investigate the role which fiber surface plays in dry soiling. These studies obviously relate to other characteristics of cotton as well. The present paper deals with the surface of fibers which have not had any chemical or additive finishing; subsequent reports will cover the effects of finishing and modification procedures, and the distribution of dry soil on both untreated and modified fibers.

The grosser details of the surface are apparent when fibers are examined with the light microscope, and many excellent photographs of the longitudinal and cross-sectional appearances of fibers exist in the literature. Study of the finer details at higher magnifications requires isolation of the surface in order to eliminate interference from the interior regions of the fiber. The technique of surface replication is very

[1] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U.S. Department of Agriculture.

useful for this purpose. Moreover, the greater resolving power of the electron microscope can be used to advantage in the study of surface replicas. The publications of Kling and Mahl [4] and of Kinsinger and Hock [3] have demonstrated the quality of results that can be expected when examining the surface of cotton by these techniques.

The surface of untreated cotton fibers might be expected to show variations with respect to inherent characteristics, such as species and variety, stage of cell wall development, cell wall thickness, or growth conditions. Similarly, effects due to mechanical forces of handling or use should introduce differences in the fiber topography, although the extent of these differences can hardly be predicted. Fibers whose characteristics and history were known to vary in these respects were examined in this study. Our observations have shown that, in general, the surfaces of native cottons have a relatively uniform appearance. Certain specimens, however, presented qualities which seemed to be related to their physical condition or history. The characteristic microscopic topography of the cotton fiber in the native state is distinctive, and would readily serve to differentiate cotton from other textile fibers.

Methods

Polystyrene film was found to be a suitable medium for obtaining replicas of fiber surfaces. A film may be formed by spreading a 5% solution of polystyrene in xylene, containing 0.5% dibutyl phthalate as a plasticizer, on a clean dry microscope slide and permitting the xylene to evaporate. The thickness of the film formed may be controlled by the amount of solution placed on the slide. For light microscopy of replicas of relatively large areas of fiber surface, a film about 0.01 in. thick was found to be satisfactory. Ten or more fibers (or small squares of fabric) are laid on the dry film; another microscope slide is placed above the specimen, and three screw clamps of the type used for constricting rubber tubing are applied to the "sandwich" thus formed. The clamps are tightened taking care not to cause cracking of the slides; the extent of tightening permissible is readily determined by experience. The assembly is placed in an oven at 50° C. for 1 hr. After removal and cooling to room temperature, it is dismantled. The fibers or fabric swatch may be grasped with tweezers and gently pulled free from the polystyrene. Short lengths of cotton which occasionally remain attached may be dissolved with cuprammonium hydroxide, but in most cases this procedure is not necessary.

The fiber impression obtained in this manner, using a thick film of polystyrene, is usually suitable for examination with the light microscope without further treatment. If greater contrast is desired, the surface irregularities may be emphasized by evaporating gold on the replica from an oblique angle, in the manner routinely used for "shadow-casting" in electron microscopy.

Polystyrene replicas of sufficient thinness to be examined in the electron microscope were found to be mechanically weak and difficult to manipulate successfully. A "two-stage" process for preparation of replicas, in which the initial impression ("negative") is reproduced in a second film ("positive") was found to give satisfactorily strong replicas. The positive is formed by the vertical evaporation of silicon monoxide or better, of carbon, on to the negative polystyrene impression. The thickness of this positive film is approximately 200 Angstrom units. The polystyrene layer, which is made much thinner than in the preparations for light microscopy, is removed from the positive layer by placing the double film, polystyrene side up, on a specimen grid, and allowing the grid to rest on filter paper saturated with xylene or a 1:1 mixture of carbon tetrachloride and methyl ethyl ketone. Ordinarily the polystyrene is completely dissolved by overnight exposure to this solvent mixture. The positive carbon or SiO replica remaining is shadowed with an appropriate metal to emphasize irregularities present, and examined in the electron microscope. The width of the fiber replica is usually about 10 microns, i.e., roughly one-fourth of the fiber perimeter is replicated. Obviously, only a small fraction of the total fiber surface can be examined in this manner.

Most of the specimens studied were examined by both light and electron microscopy. The two types of observations are complementary, and provide orientation for more complete interpretations of the characteristics of a specimen.

Results and Discussion

Fibers from Unopened Bolls

In order to study changes which take place in the surface of fibers when the boll opens and the cotton dries for the first time, fibers were taken from an unopened boll whose age (from flowering) was

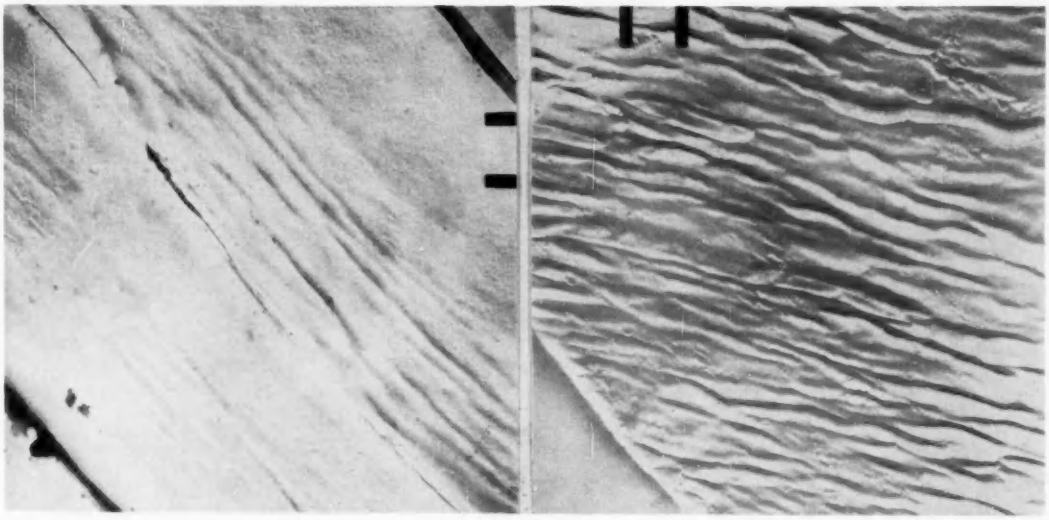

Fig. 1. Electron micrographs of surface replicas of fibers from an unopened boll before (left) and after (right) drying for the first time. Polystyrene-carbon method. Distance between markers is 1 micron.

known to be 54 days. Surface impressions were made immediately after removing the fibers from the boll, while they were still wet with boll moisture. Other fibers from the same boll were allowed to dry in air and their surfaces replicated. Figure 1 shows the topography of the fibers in these two conditions. It is apparent that the surface of the undried fiber is considerably smoother than that of its dry counterpart. This observation is to be expected, since it is known that there is a marked decrease in the cross-sectional area when such fibers are dried [1]. Such shrinking would bring about folding and wrinkling of the cell wall, increasing the roughness of the surface boundary. Wetting of the dried fiber does not restore the original smoothness, or even decrease the roughness to any noticeable extent. The increase in cross-sectional area when dry cotton is wet amounts to about 30% [5], which appears to be considerably less than the decrease accompanying the initial drying of the fiber [1].

Fibers of Differing Cell Wall Development

The extent to which a fiber will shrink on drying should be determined, in part, by the thickness of secondary wall present. If the primary wall membranes in both thick-walled and thin-walled fibers of the same variety have the same dimensions, more wrinkling of the membrane of the thin-walled fiber should be expected when the fibers are dried. Fibers from the same sample of American Upland (Empire variety) were separated into thick- and thin-walled groups after being differentially dyed by Goldthwait method [2] in order to facilitate their distinction. Fibers from each group were selected which were clearly representative of extremes in cell wall thickness, and their surfaces replicated. Figure 2 shows the appearance of typical surfaces of thin- and thick-walled fibers. No striking differences in the over-all topography were apparent in any of the replicas examined in this experiment. It may be concluded that the shrinkage of both types of fibers is large enough to produce similar degrees of wrinkling and folding of the surface.

Fibers of Different Species

Samples of native fibers of three species prevalent in commercial cottons were examined. *Gossypium barbadense* was represented by Egyptian Karnak and Sea Island varieties. A number of American Upland cottons were chosen as typical of *G. hirsutum*. Samples of cottons grown in India (Sind) and China (Tientsin) were selected as specimens of *G. herbaceum*. The topography of each species of cotton was generally similar to that of the others. Typical micrographs obtained from examination of the surfaces of Egyptian Karnak, Deltapine (Upland) and

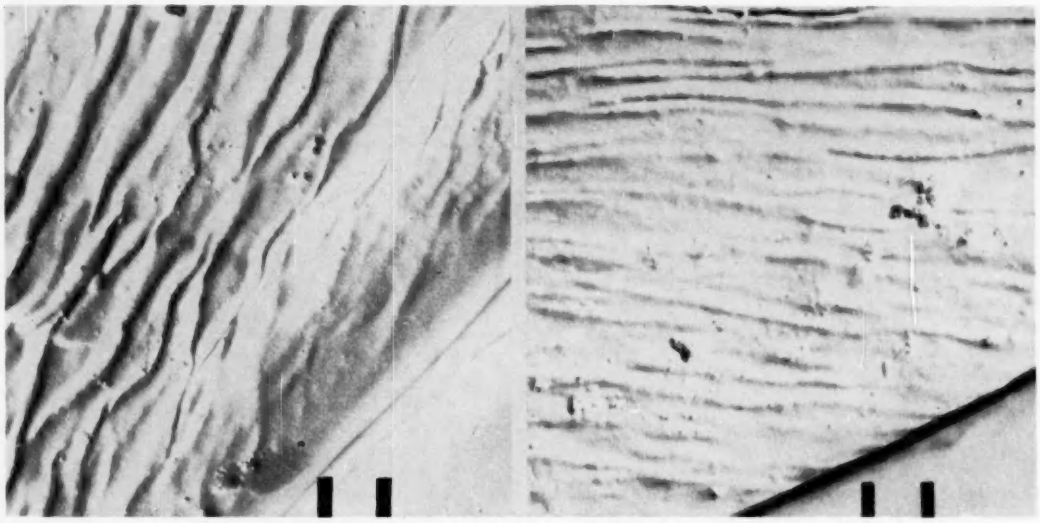

Fig. 2. Electron micrographs of surface replicas of thin-walled (left) and thick-walled (right) Empire cotton fibers. Polystyrene-carbon method.

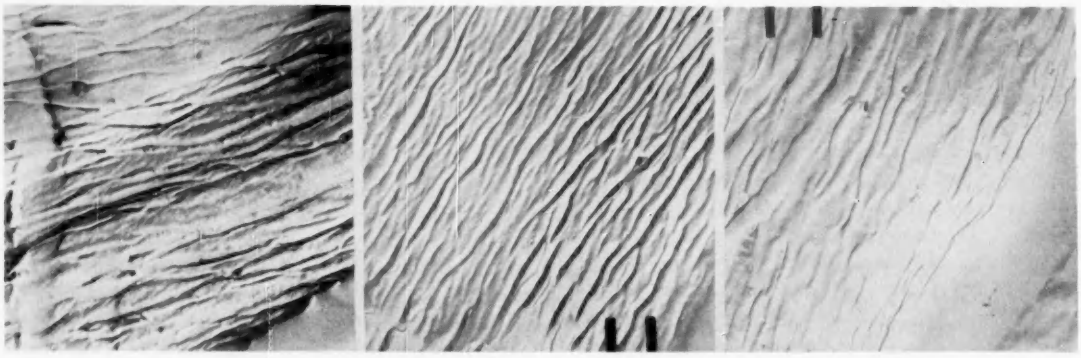

Fig. 3. Electron micrographs of surface replicas of Egyptian Karnak (left), Deltapine (center) and Chinese Tientsin (right) cotton fibers. Polystyrene-carbon method.

the Chinese cotton are seen in Figure 3. It is doubtful that cotton species could be distinguished on the basis of the appearance of their surface replicas.

Fibers of Different Variety

The general similarity of the surface patterns of cottons of different species also held true for various specimens of varieties of *G. hirsutum* and related cottons. Samples of Deltapine, Rowden, and Acala varieties, each grown under irrigation and under conditions of natural rainfall, were examined. Other samples studied included Empire, Arkansas Green Lint, and an experimental Acala cotton which had especially high luster. The Green Lint cotton surface was somewhat smoother than that of the others. The lustrous experimental Acala sample showed no surface characteristics by which it could be distinguished from most other cottons.

Qualitative and Quantitative Aspects of Fiber Topography

The qualitative similarity of most of the fiber surfaces examined by the replica technique stems from the essentially identical architecture of each fiber.

The pattern of roughly parallel ridges and grooves spiraling at an oblique angle (usually 20–30°) to the fiber axis is common to all native cotton surfaces. This pattern is undoubtedly determined by the spiral arrangement of the cellulose fibril groups which lie immediately beneath the primary wall membrane. Direct evidence of this was afforded by preparing a replica of the fiber surface at the regions where reversals of the spiral direction of the secondary wall fibrils occurred. The position of these areas is readily determined by polarized light microscopy. The change in direction of the surface grooves is illustrated in the photomicrograph of a surface replica in Figure 4. In a distance of approximately 100 microns, two reversals are seen. Finer details of the fiber surface in the region of a reversal are shown in Figure 5. The ridge-and-groove system in the area pictured is parallel to the fiber axis for a brief interval only, and quickly assumes the typical direction observed in most micrographs.

Estimation of the angle which the ridges make with respect to the fiber axis showed no consistent value which could be associated with the sample under study. This result was not unexpected, in that the replicated areas of the fiber are only a small fraction of the total surface, and considerable variation in orientation on such a small scale is not unusual in materials like cotton. Also, since the angle of the ridges would be determined largely by the arrangement of the cell wall immediately beneath the fiber surface, any close relation to the over-all orientation of the fibrils in the interior of the fiber cannot be presumed. The measured angles in native fibers ranged from 0° to a maximum of 45°, with an average value of about 30°.

An investigation was made of the possibility that the ridge pattern on the surface of cotton might show a variation that could be associated with the sample type. The distance between ridges was measured by projecting micrographs of surface replicas on a screen at known magnification. The number of ridges present in a given distance (measured normal to the general direction in which the ridges lay) was counted. This procedure was repeated on as many areas of the sample as had been photographed. The results are listed in Table I, in which 14 types of cotton are arranged in order of increasing average distance between surface ridges. The number of measurements listed represents that number of different replicated areas of each sample.

It is apparent that the cottons studied did not show an extremely large difference in the average distance between ridges, the range being from 0.38 to 0.68 micron, although statistical analysis showed that the variations noted were often significant [6] at the 1% confidence level. Table I lists, for each sample,

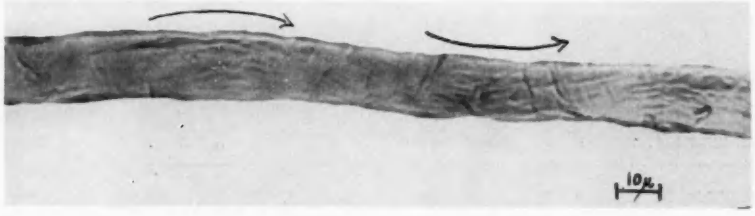

Fig. 4. Photomicrograph of surface replica of fiber showing reversals in the direction which grooves make with respect to fiber axis.

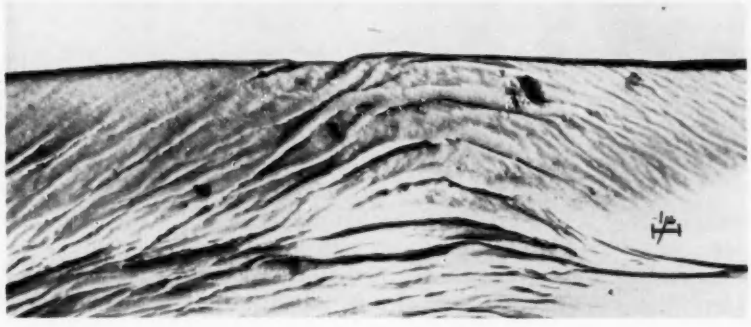

Fig. 5. Electron micrograph of surface replica of fiber showing reversal in direction of ridge-groove system. Polystyrene-carbon method.

TABLE I. Average Distance between Ridges on Surface of Cotton Fibers

Sample No.	Sample description	Number of measurements	Mean distance between ridges, micron	Standard deviation, micron	Sample numbers for which means are significantly different at 1% confidence level
1	Deltapine (irrigated)	180	0.38	0.13	—
2	Rowden (irrigated)	48	0.42	0.07	1
3	Deltapine (rain-grown)	74	0.46	0.09	1–2
4	Acala (irrigated)	73	0.47	0.09	1–2
5	Chinese Tientsin	19	0.49	0.09	1–2
6	Acala ("high luster")	14	0.49	0.07	1–2
7	Egyptian Karnak	76	0.51	0.17	1–2
8	Rowden (rain-grown)	73	0.52	0.09	1–4
9	Empire (thin-walled)	33	0.55	0.11	1–4
10	Deltapine (mercerized)	48	0.56	0.14	1–4
11	Acala (rain-grown)	87	0.58	0.09	1–8
12	Rowden (mercerized)	50	0.58	0.14	1–6, 8
13	Empire (thick-walled)	20	0.61	0.09	1–8
14	Scoured cotton	18	0.68	0.21	1–8

those samples which differed significantly. For example, Deltapine cotton grown under irrigation was found to have a significantly lower distance between ridges than any of the other cottons measured.

The thick-walled fibers of Empire cotton showed a larger average distance between ridges (0.61 micron) than the thin-walled fibers obtained from the same sample (0.55 micron). Perimeter measurements on similar fibers indicate that the thick-walled fibers have a perimeter about 10% greater than the thin-walled. If the perimeter of the primary membrane is considered to be approximately constant for a given variety, it follows that the number of folds, i.e., ridges, would be fewer on the surface of a fiber whose interior was more completely filled.

With respect to species, the differences noted are not clear-cut. Deltapine (rain-grown) (*G. hirsutum*) had a smaller average distance between ridges than Chinese Tientsin (*G. herbaceum*) or Egyptian Karnak (*G. barbadense*), but the latter two showed no significant difference. The wide variation in fineness in three cottons indicates that this property does not directly affect the surface pattern of the fibers. The Egyptian and Chinese cottons represent the extremes in fineness in the samples examined in this study.

In the American Upland varieties measured, significant differences were noted between Deltapine and Rowden varieties grown under rainfall. Consistently smaller distance between ridges was found for cottons grown under irrigation when compared with the same varieties grown under rain. Mercerization and scouring appeared to increase the distance between ridges.

Examination of the surface replica micrographs shows that the distance between individual ridges varies greatly. The standard deviation of the distances measured emphasizes this point. Although the possibility of using such measurements as a means of distinguishing the species or variety of native cotton samples appears to exist, the large inherent errors of sampling and technique would require extensive critical testing of the method before its validity could be established.

The depth of the grooves is likewise extremely variable, but appears to average 0.5 micron. Their depth has been observed occasionally in cross sections of cotton examined in the electron microscope. Another method for estimating depth is the observation of the position of particles of known size which have settled in the grooves. The ridge system covers virtually every area of the fiber surface, and many individual ridges appear to be 10–20 microns or more in length.

Effects of Mechanical Handling

It might be expected that tension, compression, or abrasion during handling would effect changes in the surface of cotton. Fibers subjected to each of these types of treatment were examined. Tension did not appear to cause any detectable difference in the surface characteristics of the fibers. In these experiments, tension forces exceeding 2 g. per fiber could

not be applied during the replication procedure, as rupture invariably occurred.

Fibers taken from portions of fabric known to have been in compression during use showed characteristic surface formations. In such fibers, there were regions having one or more narrow bulges extended out from the main plane of the fiber surface, running transversely to the fiber axis. An example of this formation is shown in the micrograph in Figure 6. Occasionally, fibers which had not been knowingly exposed to compressive forces also possessed this formation. The phenomenon was produced in fibers under longitudinal compression as well as multidirectional, i.e., hydraulic, compression. The origin of the formation probably lies in the irreversible buckling of the cellulose fibril structures beneath the fiber surface.

Untreated fibers, even after ginning, opening, and carding, seldom showed tears or breaks in the continuity of the surface layer. Violent abrasion, as might be undergone by fibers on cloth buffing wheels, causes breakdown and removal of the surface layer, so that the characteristic topography is completely changed. An example of a strongly abraded surface is shown in Figure 7, in which the replicated fiber was taken from a buffing wheel. Particles of metal or other hard material are seen embedded in the fiber, having been transferred to the replica during its preparation.

Conclusions

The outstanding characteristic of the surface of native cottons from widely different sources is the pattern of minute ridges spiraling around the fiber. While it cannot be shown that this pattern is of importance in determining the macroscopic behavior of cotton, its presence directly affects phenomena at the microscopic level. The innumerable ridges and grooves offer opportunity for the attachment of small-sized alien particles which would otherwise show little tendency to concentrate on the fiber surface. The roughnesses observed demonstrate that the cotton surface is undulatory in character; the periodicity and amplitude of the undulations are of the order of 0.5 micron. The customary microscopical estimations of the perimeter and surface area of fibers, which do not take into account this small-scale deviation from planarity, will thus give results which are lower than the actual dimensions existing in the fibers. An estimate of perimeter which allows for the profile of the ridge-groove system shows that the actual perimeter of the fiber is about twice that calculated for a smooth profile.

Fig. 6. Electron micrograph of surface replica of fiber showing formation apparently produced by longitudinal compression. Polystyrene-carbon method.

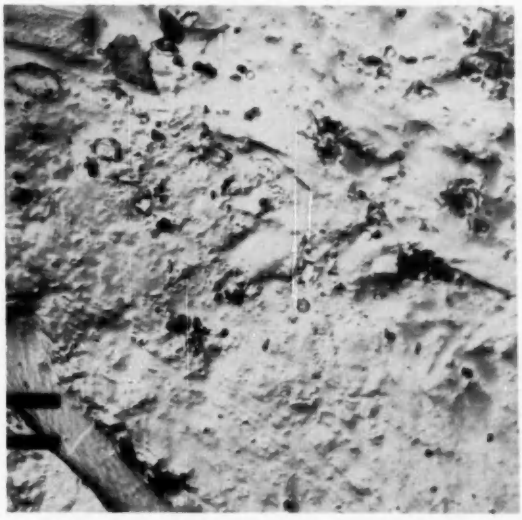

Fig. 7. Electron micrograph of surface replica of abraded fiber taken from buffing wheel cloth. Polystyrene-carbon method.

It is clear that the surfaces of all commercial cotton fiber possess some degree of wrinkling. The only notably smooth surface exhibited was that of fibers removed from an unopened boll while still wet. While differences in luster conceivably could arise from variations in fiber topography, it appears that these differences are produced by other factors. A cotton with a smooth surface could be advantageous in certain respects, e.g., reduction of readily accessible surface area. However, the roughening of the surface arises mainly from the loss of a large volume of water which is an integral part of the growing fiber. Reduction of the accompanying shrinkage would be extremely difficult to achieve within the framework of current cotton production methods.

The relative frequency with which the ridges occur on the fiber surface appears to differ from one type of cotton to another. These differences were significant in many of the samples examined in this study. It must be borne in mind, of course, that cotton samples of the same variety display wide variations in physical properties as a result of differences in environment during growth. It is probable that similar differences would be found in quantitative evaluations of the surface pattern of such materials.

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The Surface of Cotton Fibers

Part II: Modified Fibers

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Abstract

Microscopical studies of surface replicas of chemically finished or modified cotton fibers have shown that the characteristic surface of native cotton is often altered by various commercial and experimental treatments. While removal of the wax from the fiber surface has little effect, scouring usually uncovers the fine cellulose fibrils of the primary or even secondary walls. Mercerization does not eliminate the rugosities of the native fiber surface. Additive finishes, including starch, carboxymethyl cellulose, colloidal silica, and acrylic polymer resins change the original fiber topography to an extent dependent on the amount applied. In general, particulate additives tend to concentrate in the grooves of the fiber surface when applied in dilute formulations, but cover most of the fiber surface at higher concentrations.

Chemical modifications which increase the fiber cross-sectional area make the fiber surface smoother, but extensive swelling of the cellulose derivative during its preparation appears to create roughness in some modifications. Crease-resistant finishes do not alter the fiber topography significantly.

IN Part I of this series [11], it was shown that the major characteristic of the topography of native cotton fibers is a pattern of parallel ridges and grooves spiraling around the fiber at an acute angle to its axis. The present paper deals with the surface features of fibers which have undergone finishing or chemical modification. The striking changes in the behavior of modified cottons due to these treatments would lead one to predict that fiber topography is profoundly affected by such treatments. For many of the modified cottons examined in this study, this is indeed the case. As the studies of Kling and Mahl [3, 4] have shown, scouring and enzymatic attack strongly affect the fiber surface. Additive and modification finishing operations practiced commercially and experimentally have received less attention in this respect. Knowledge of their effects is important in considering various aspects of the processing and end uses of cotton textiles, as surface is a factor in fiber behavior in virtually all environments.

The methods of microscopical examination by the replica technique have been described [11]. The specimens of modified cotton were obtained, for the most part, from samples prepared in this laboratory.

[1] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U.S. Department of Agriculture.

Purification Treatments

Most cotton textiles receive treatments designed to remove a portion or all of the noncellulosic substances present in the native fiber. Such substances interfere with subsequent finishing procedures, and detract from the desired whiteness of the textile. Noticeable changes in the surface behavior of cotton as a result of such purification indicates that the outer layers of the fiber have been strongly affected, and an examination of fiber surfaces after such treatments was carried out.

Removal of Wax

Native fibers were extracted with ethyl alcohol in a Soxhlet apparatus for 4 hr. to remove the waxy materials present. The temperature of the alcohol in the chamber was approximately 60° C., and 8 passes of solvent occurred during the extraction period. The extracted fibers were rinsed with water and allowed to dry in air. They wet readily with water after drying, in contrast to the slow wetting of the native

fibers. A replica of the surface of an extracted fiber, prepared by the polystyrene-SiO method [11], is shown in Figure 1. In agreement with the findings of Kling and Mahl [3] on similarly treated material, the characteristic ridge pattern of the native fiber surface was retained. The ridges themselves were somewhat sharper, but no other differences were obvious. The increase in sharpness of the ridges may be the result of wax removal, which is virtually complete in the method employed. No evidence of the cellulosic fibril system of the primary wall was present; materials other than wax undoubtedly remain after alcohol extraction and obscure the cellulose pattern.

Scouring and Bleaching

Fibers from fabrics which had been commercially scoured and bleached were also replicated and examined. Previous studies [10] of the effect of scouring with hot dilute NaOH on the primary wall of cotton have shown that there is a progressive removal of wax, pectic materials, and other noncellulosic substances. If carried far enough, the network of cellulose fibrils which is the framework of the primary wall is revealed. Bleaching with hypochlorite or hydrogen peroxide subsequent to scouring did not appear to remove more noncellulosic material. Kling and Mahl [3], who had studied replicas of whole fibers subjected to various scouring conditions found similar results. The typical appearance of a commercially scoured and bleached fiber surface is shown in Figure 2. Such purification did not eliminate the ridges present on the surface of the untreated fiber, but their frequency and sharpness was reduced. Other commercially scoured and bleached fabrics, of whiteness and appearance equal to that of the fabric from which Figure 2 was prepared, were scarcely distinguishable from unscoured cottons in the details of their surfaces. Considerable variation in the amount of noncellulosic materials removed by commercial methods is indicated.

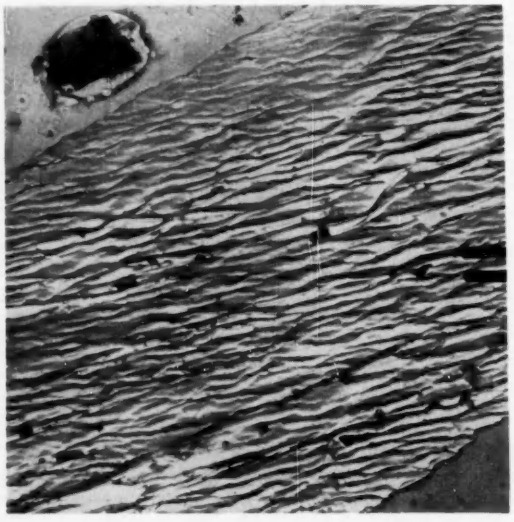

Fig. 1. Electron micrograph of surface replica of fiber extracted with hot ethyl alcohol. Polystyrene-SiO method. Distance between markers is 1 micron.

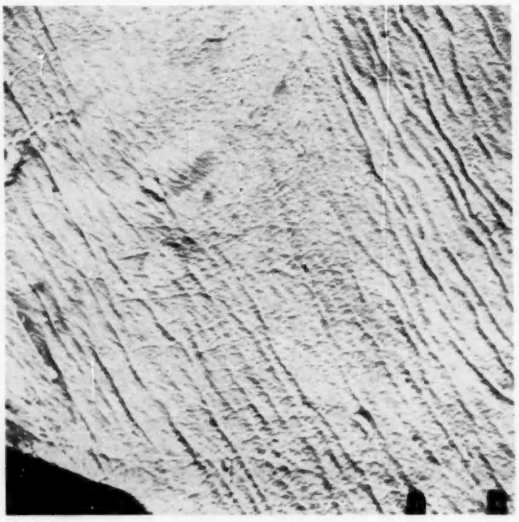

Fig. 2. Electron micrograph of surface replica of fiber from commercially scoured and bleached fabric. Polystyrene-carbon method.

Some specimens of scoured and bleached cotton exhibited regions of fiber surface where the primary cellulose network was ruptured and pushed back, exposing the secondary thickening of the fiber. An example of this situation is shown in Figure 3, where pieces of primary wall cellulose are seen to be pulled aside and crumpled up, while the highly parallelized microfibrils of the secondary wall are prominently displayed. Kling and Mahl [3] attribute the tearing and removal of the primary wall to drastic methods of purification, and regard such an effect as undesirable.

The loss of wax and pectic materials from the surface layer of cottons obviously changes the chemical

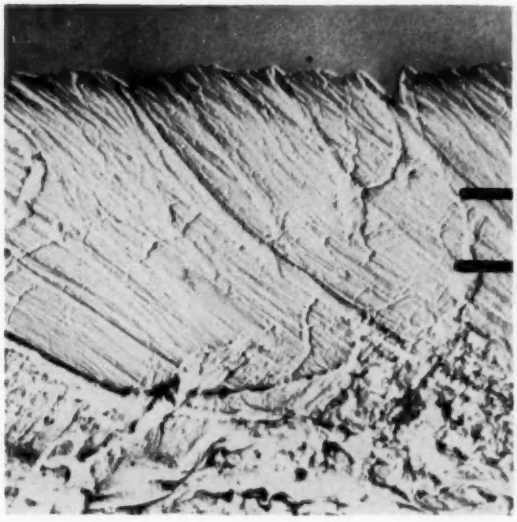

Fig. 3. Electron micrograph of surface replica of commercially purified fiber exhibiting rupture of the primary wall cellulose network and exposure of secondary wall cellulose. Polystyrene-carbon method.

nature of the fiber surface. The appearance of a fibrillate texture after scouring also alters the surface of such materials from a topographical standpoint, although the ridges of the native cotton are retained. Measurement of the average distance between ridges on fibers from a scoured American Upland cotton fabric was 0.68 micron (mean of 18 observations); this value is significantly greater than those measured for most native fibers [11]. While reduction of the number of rugosities by scouring would lead one to conclude that such fibers would soil less readily, it was found that scoured fibers soil to an appreciably greater extent than do native fibers. This tendency must arise from the creation of innumerable minute pores in the surface of the fiber by removal of wax and pectic materials. Similarly, scoured fibers are more permeable to liquids and reagents. These changes undoubtedly increase the accessible area on the surface of the fiber; a structure of the type shown in Figures 2 and 3 has more surface area available for contact with extremely small particles than does a native fiber surface. The concomitant changes in the chemistry of the surface are also of decisive importance in altering the response of the fiber in such environments. The untreated primary wall which is the outermost layer of the fiber has been shown to be approximately 50% cellulose [10]; in the scoured fiber the cellulose content is probably well over 90%.

Mercerization

The striking changes in the cross-sectional shape of cotton fibers which occur on mercerization [5] would lead to the surmise that the fiber surface would also be affected strongly by such treatment. A number of fiber samples which had been mercerized in different ways were selected for surface study. These included native fibers treated in the laboratory with 20% NaOH solution for several minutes at room temperature, some being allowed to shrink freely in the caustic solution while others were held at constant length; commercially mercerized fibers from fabrics were also examined. The fibers mercerized in the laboratory were washed with dilute acetic acid and with water to remove the alkali before drying and replication. Figure 4 shows the typical microscopic topography of fibers mercerized with free shrinkage and under restraint. A comparison of these surfaces with those of untreated cotton makes it clear that neither condition of mercerization has eliminated the characteristic rugosities of the fiber, although there is some indication that the height of the ridges has been reduced. Considering the removal of the most of the gross concavities of the fiber cross-sectional shape by mercerizing, it is somewhat surprising that the mercerized fiber surface is not much smoother than that of the native. The appearance of the surface of fibers from commercially mercerized fabrics was similar to that of those mercerized in the laboratory.

Microscopical observations made during mercerization indicated that the fiber cross-sectional area increases about 100% when the fiber is placed in contact with 20% NaOH. The primary and secondary walls come into intimate contact as a result of pressure developed within the fiber. Bonding between the two walls is thus enhanced, and the primary wall is difficult to remove from mercerized fibers by mechanical means [10]. The roughness of the surface of mercerized cotton is, then, a reflection of the pattern of the secondary wall structures beneath it.

The relatively small change in surface appearance brought about by mercerization is, of course, accompanied by a pronounced increase in the luster of cotton fibers. Apparently the fiber surface, as such,

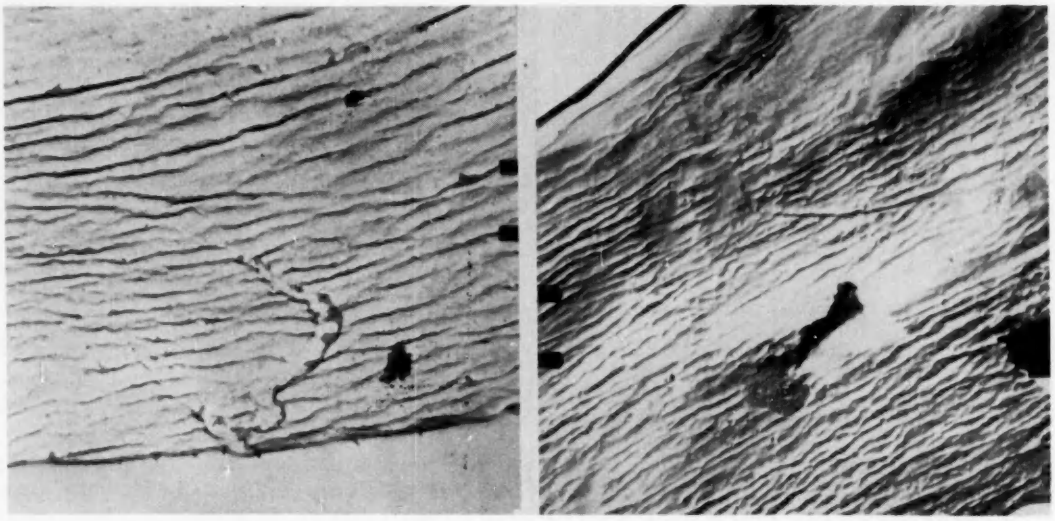

Fig. 4. Electron micrographs of surface replicas of fibers mercerized without tension (left) and at normal length (right). Polystyrene-carbon method.

is not one of the more important factors contributing to luster in cotton.

Measurement of the average distance between ridges on two samples of native cotton mercerized without tension showed a significant increase after mercerization [11]; the change for Deltapine cotton was from 0.46 micron in the native state to 0.56 micron in the mercerized state. Corresponding figures for Rowden cotton were 0.52 micron and 0.58 micron. There was some indication that mercerization under tension reduces the average angle at which the ridges lie with respect to the axis, but the large variability observed did not permit reliable estimation of such a change.

The network of microfibrils present on the surface of scoured cotton was not in evidence in such fibers after mercerization. A similar observation was made in the case of primary wall membrane which had been mercerized after being freed from the fiber body [10]. The swelling of the cellulose during mercerization apparently produces a "welding" of the microfibrils, resulting in a complete loss of discreteness of the individual fibrillar units of the primary wall.

Decrystallization

The treatment of cotton with ethylamine and other aliphatic amines which produce intramicellar swelling superficially resembles mercerization in that swelling and shrinkage of the fiber accompany the addition and removal of the amine. The crystallinity or lateral order of the cellulose is reduced to a considerably greater extent in such treatments than in mercerization [9]. The sample of cotton chosen for surface replication was a scoured yarn which had been exposed to essentially pure ethylamine overnight at 17° C. and the amine removed by extraction with hot chloroform. The surface appearance of the fibers from this sample was in all respects similar to that of untreated scoured fibers.

Additive Finishes

Necessary or desirable modification of the properties of yarns and fabrics can often be achieved by the application of high polymeric or particulate materials to cotton textiles. In most cases there is no good evidence that such materials penetrate the cell wall of the fibers to any great extent. Additive finishes, being confined to the fiber surface, would thus be expected to change the topography of cotton to a marked extent.

Starch Sizing

The application of starch for warp sizing or for stiffening of fabrics is widely practiced. The surfaces of two types of samples on which starch had

been deposited were examined. A warp yarn from a 48×48 sheeting sized with a commercial preparation to give 7% dry pickup of sizing on the fabric (14% on warp yarn) was replicated. Figure 5 shows typical results obtained for this sample. The thickness of the starch deposit was variable, and some areas did not show a discernible amount of starch. Some of the depressions in the surface of the fiber have been filled by the sizing material. Fabrics to which household starch had been applied from water dispersion to give 2-4% dry pickups were also studied. In pickups at this level, the presence of starch was not evident in most fibers, and their over-all appearance was similar to that of untreated fibers. When a dilute starch suspension was allowed to dry on a smooth silicon monoxide film, electron microscopical examination of the residue showed that the starch takes the form of irregular flat deposits of varying thickness. It seems probable that the distribution of starch on cotton surfaces is also relatively uneven at the microscopical level.

Carboxymethyl Cellulose

A "low viscosity" commercial grade of carboxymethyl cellulose (CMC) was applied from water solution to lightweight cotton fabrics to give dry pickups in the range 0.5 to 4%. Deposits of CMC at these levels have been shown to be effective in improving the ease with which soil is removed from cotton in laundering. At the lower range of pickups, the presence of CMC was not apparent in the surface replicas prepared, but at the 4% level, the fibers had the appearance shown in Figure 6. In this sample, taken from an unscoured fabric to which 4% CMC had been applied, the additive finish seemed to form a continuous film on the surface of the fibers. Small crevices were filled, but the over-all topography of the cotton was not changed. On scoured fiber surfaces, the fibrils were coated, but were still readily discernible in samples with 4% pickup.

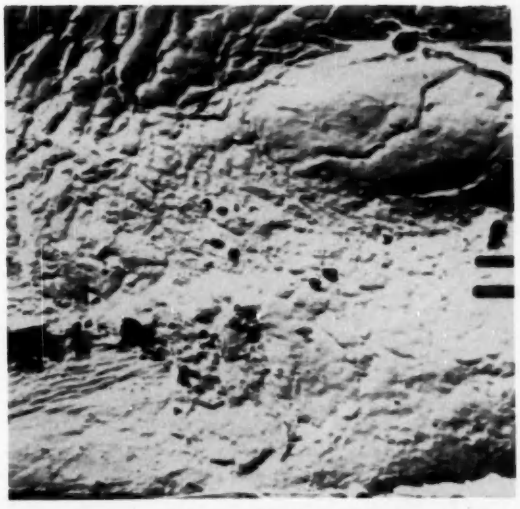

Fig. 5. Electron micrograph of surface replica of fiber in starch-sized warp yarn. Polystyrene-SiO method.

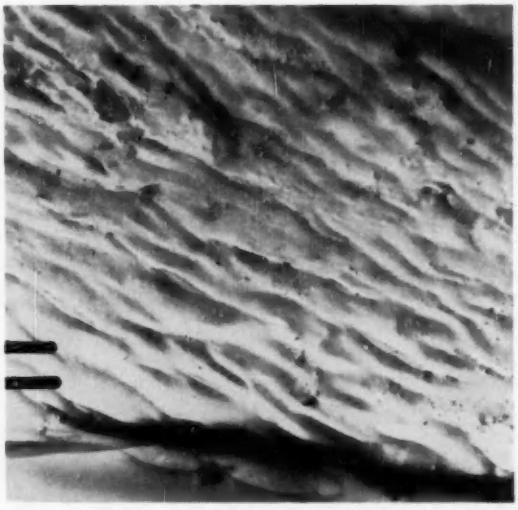

Fig. 6. Electron micrograph of surface replica of fiber from fabric to which 2% carboxymethyl cellulose had been applied. Polystyrene-carbon method.

Colloidal Silica

Particulate materials have been used to modify the frictional characteristics or soiling tendency of cotton, properties which stem directly from the condition of the fiber surface. Colloidal silica, which in practice is probably the most widely used of such materials, was applied to cotton fabrics by padding from aqueous suspensions whose concentration was adjusted to give a range of dry pickups from 2 to approximately 7%. The primary particle size of the silica used was of the order of 0.02 micron. The presence of silica particles on the surface of the fibers was evident in the replicas prepared from even the most dilute dispersions. Figure 7 shows the surfaces of fibers taken from fabrics with 3 and 7% add-ons of silica, respectively. At the lower pickup, much of the char-

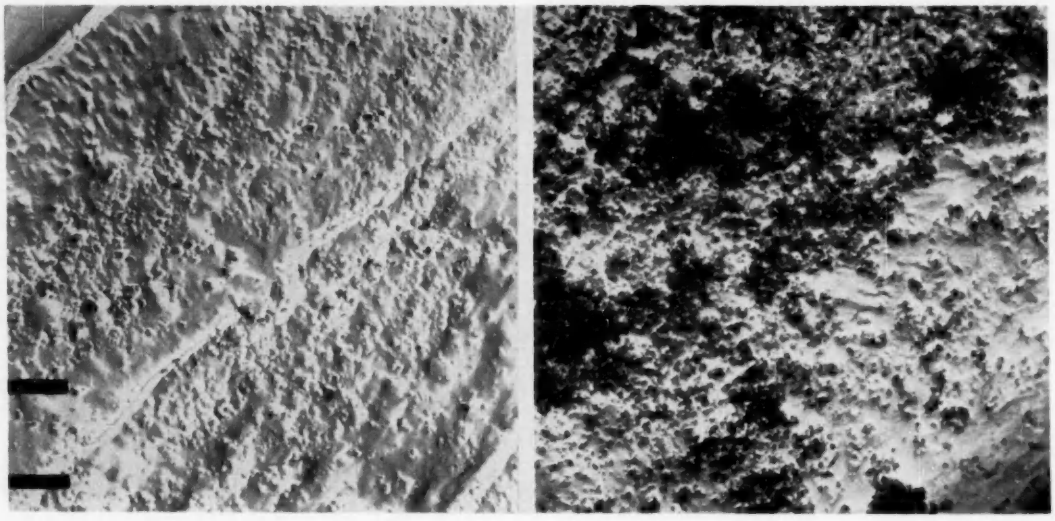

Fig. 7. Electron micrographs of surface replicas of fibers from fabrics with 3% (left) and 7% (right) pickup of colloidal silica. Polystyrene-carbon method.

acteristic contour of the fiber surface is apparent, and the silica is present mostly in the surface depressions. This observation was also the case in fibers having lower pickups. At the 7% level, the original surface is almost completely obscured by the heavy deposit. Obviously the hand and frictional properties of textiles with such deposits would differ from those of untreated material.

Polymer Lattices and Related Materials

Synthetic polymeric substances are also deposited on textile surfaces in order to modify abrasion resistance, water repellency, luster, and other properties. A number of such materials were applied to cotton fabrics in this study, including two acrylic polymer lattices and a silicone resin. Rhoplex SR (Rohm & Haas, Inc., Philadelphia, Pa.)[2] was deposited from water suspension of 80 × 80 print cloth to give a 5% dry solids pickup, being allowed to dry at room conditions. Figure 8 shows the replicated surface of fibers from this fabric, and illustrates the tendency of the relatively hard particles of Rhoplex SR to collect in the surface grooves of the fiber. The average diameter of the particles is of the order of 0.1 micron, and they are seen to fit into the surface depressions very comfortably. The particles of this material remained discrete, with no tendency to film-formation, although larger aggregates formed a layer of close-packed spheres.

The other acrylic resin application examined was Hycar 4501 (B. F. Goodrich Chemical Co., Cleve-

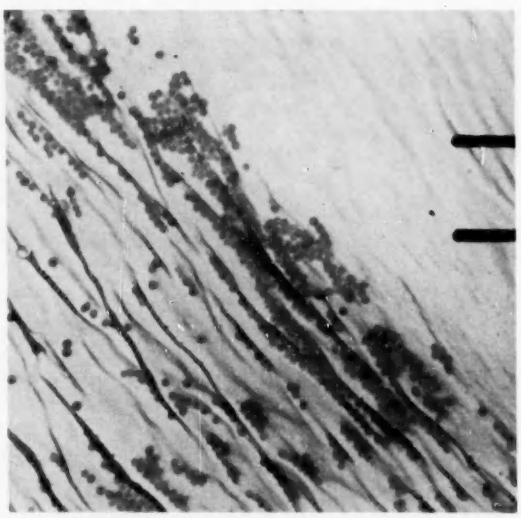

Fig. 8. Electron micrograph of surface replica of fiber from fabric treated with hard acrylic polymer latex dispersion (5% pickup). Polystyrene-carbon method.

[2] Mention of trade names does not imply endorsement of the product over similar products of other manufacturers.

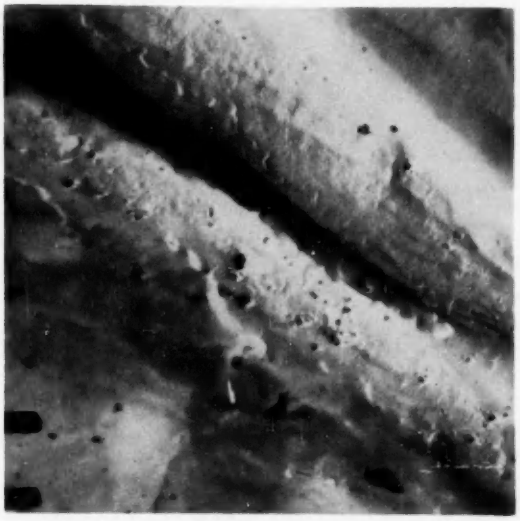

Fig. 9. Electron micrograph of surface replica of fiber from fabric treated with soft acrylic polymer latex dispersion (30% pickup). Polystyrene-carbon method.

land, Ohio),[2] a latex of a polymerized acrylic ester, which was deposited on fabric to give 30% dry solids pickup and allowed to dry at room conditions. A surface replica obtained from this fabric is shown in Figure 9. The relatively soft particles of polymer in this latex appeared to coalesce on the surface of the fiber, obliterating the details of the original surface.

The silicone water repellent finish examined was Decetex 104 (Dow Chemical Co., Midland, Michigan).[2] It was applied from emulsion by padding to the fabric to give a 2% pickup of solids, and cured according to the manufacturer's directions. The surface of fibers from this fabric showed the presence of a film which made the topography smoother than that of the original fiber, although its continuity was interrupted by formations resembling small craters.

Chemical Modifications

Most chemical modifications of cotton are aimed at achieving changes which extend beyond the surface to the internal regions of the fiber. While the reactions seldom involve every cellulose molecule or anhydroglucose unit of the fiber, it is believed that chemical modification of the internal surfaces of the fiber is usually necessary to bring about optimum effects. The addition of new groups in the inner regions of the fiber causes physical enlargement of the cross-sectional area of cotton, which is reflected in changes in the folds present on the fiber surface. Typical fibrous cotton cellulose derivatives were examined in this survey, including the partial acetate, the cyanoethyl, carboxymethyl and beta-propiolactone ethers, and the reaction product with melamine-formaldehyde.

Partial Acetylation

The esterified cotton samples examined were lightweight square-woven fabrics which had been scoured prior to acetylation. The method of acetylation was that described by Goldthwait *et al.* [2], using a mixture of acetic acid and acetic anhydride, and the acetyl content varied from 17 to 27%. This corresponds to the degree of substitution of approximately 1, the range which confers resistance to heat and enzymatic degradation. The typical appearance of the surface of partially acetylated fibers is shown in Figure 10. The fiber surface shown in this figure was obtained from a fabric containing 25% acetyl. All of the acetylated fiber surfaces were smoother than those of native, scoured, or mercerized fibers, with a marked decrease in the frequency and height of the ridges present. In the samples examined, there was no evidence of the fibrillate texture of the scoured fiber surface which was characteristic of the fibers from which the acetylated samples were prepared.

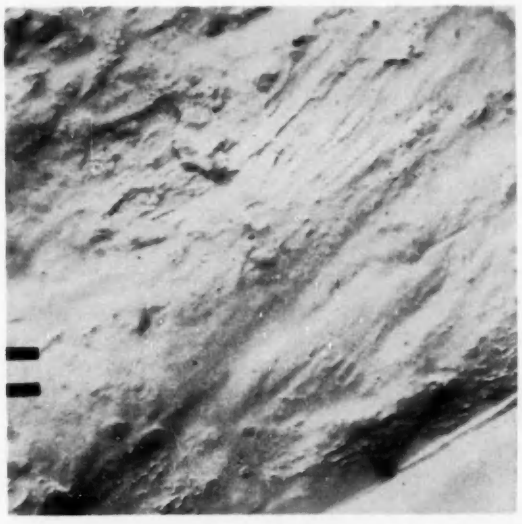

Fig. 10. Electron micrograph of surface replica of partially acetylated cotton fiber containing 25% acetyl, prepared from scoured cotton. Polystyrene-carbon method.

The disappearance of the fibrils may have occurred as a result of the swelling and solubilization of the derivative in the reagent used for esterification.

Partial Cyanoethylation

The sample of cotton etherified with acrylonitrile selected for replication was an 80 × 80 scoured print cloth containing 4% nitrogen. It was prepared as described by Daul *et al.* [1], using 2% NaOH as catalyst, purified acrylonitrile, and a reaction temperature of 60° C. The degree of substitution was approximately 0.5, which is high enough to impart rot resistance to the cellulose substrate. Figure 11 shows the replicated surface of a fiber from this fabric. Although the over-all topography of the fibers was somewhat smoother than that of the untreated (scoured) starting material, poorly defined fibrils were still present in both primary and secondary wall arrangements. As in partial acetylation, the increase in cross-sectional area of the fiber and the sensitivity of the reacted cellulose resulted in a fiber whose surface differs from that of untreated cotton.

Partial Carboxymethylation

The introduction of bound carboxymethyl groups into cotton, in contrast to the surface deposition of CMC, yields a stable textile material capable of ion-

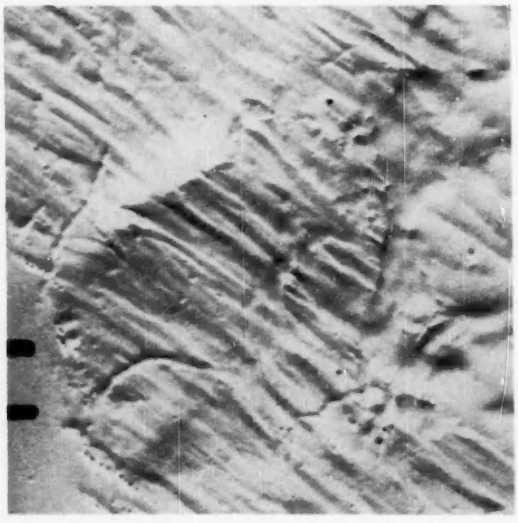

Fig. 11. Electron micrograph of surface replica of partially cyanoethylated cotton (4% nitrogen). Polystyrene-carbon method.

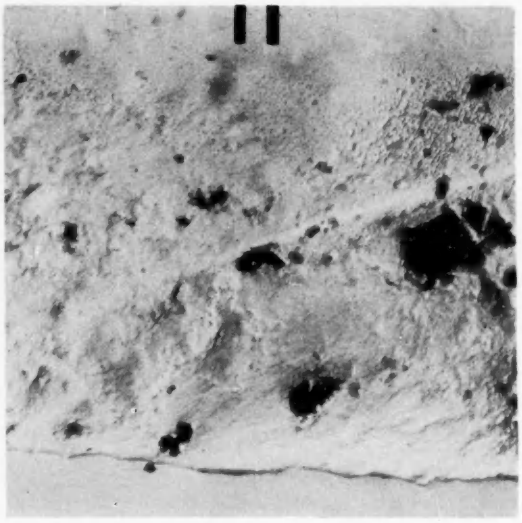

Fig. 12. Electron micrograph of surface replica of partially carboxymethylated cotton fiber. Polystyrene-SiO method.

exchange as well as other interesting properties [7]. The carboxymethylated cotton whose surface was replicated in this study had a degree of substitution of 0.1, which is below the level at which solubility in dilute alkali occurs. It was prepared by padding a scoured 80 × 80 print cloth with 15% monochloroacetic acid, followed by immersion in 40% NaOH; the reaction was carried out at room temperature for 0.5 hr. Subsequent acid rinsing and washing removed the excess alkali. The typical surface of the fibers in this fabric is shown in Figure 12. It was clear that the characteristic ridge pattern of the untreated fiber surface had been destroyed in this modification. The deposition of CMC on the surface of fibers did not show such a marked alteration on the fiber topography (Figure 6). In the carboxymethylated cotton, vestiges of the microfibrillate texture of the surface were present, but discrete fibrils were difficult to discern. The relatively drastic swelling conditions to which the cotton is exposed during the carboxymethylation reaction are probably responsible for the somewhat eroded appearance of the fiber surface.

Reaction with Beta-Propiolactone

Another cotton cellulose ether (or ester) of current interest whose surface was examined was the reaction

product with beta-propiolactone [8]. The samples studied were yarns which had been reacted under differing conditions to give approximately equal weight gains (20%). The surface of the fibers from the yarn which was reacted with beta-propiolactone in xylene at the boiling point was found to possess large masses of polymeric material attached, even after extraction with acetone, in which polymerized beta-propiolactone is soluble. These masses were easily visible with the light microscope. On the other hand, the sample which had reacted in the presence of sodium hydroxide to the same weight pickup showed no surface characteristics which would distinguish it from the untreated control material.

Wrinkle-Resistance Treatments

The impregnation of cotton with urea-formaldehyde, methylol melamine, and similar substances, followed by curing to cause reaction or polymerization is common to many current wrinkle-resistance treatments [6]. It is believed that cross-linking of adjacent cellulose chains occurs to some extent in such modifications. Microscopical staining techniques have shown that, in most cases, the cured resin is distributed throughout the cell wall of the fibers. Thus, at the pickups which give satisfactory results (ca. 5%), relatively little resin should be located on the fiber surface. The surfaces of the fibers in a scoured 80 × 80 print cloth which had been treated with Resloom HP (Monsanto Chemical Co., Springfield, Mass.)[2] were replicated and examined. Resloom HP, a finish of the methylol melamine type, was applied with a catalyst and cured at 150° C. for 5 min., and the weight gain in this sample was 8.2%. The appearance of a fiber surface obtained from this fabric is shown in Figure 13. The ridge pattern of the untreated fiber is still present, and remnants of the primary wall also are seen in this sample.

Fig. 13. Electron micrograph of surface replica of cotton fiber from fabric treated with methylol melamine resin (8% pickup). Polystyrene-carbon method.

Conclusions

The microscopic topography of cotton is affected to varying extents by the treatments designed to modify the surface or internal character of the textile structures. The fundamental quantitative nature of the modification achieved in many cases seems to be the determining factor in changing the original form of the fiber surface. For example, after purification treatments, where significant quantities of material are removed from the outer layers of the native fiber, the surface has an entirely new appearance. The change noted in this case is predictable and is in line with the known architecture of the fiber. Similarly, the deposition of particulate or even completely amorphous materials on the surface of cotton will produce a noticeable alteration of the fiber topography at relatively low pickups. The changes observed in additive finishing are undoubtedly made evident because of the limitation of the finish to the fiber surface. Chemical modifications which change the size of the cross section of the fiber significantly also affect the surface. Presumably, permanent physical enlargement of the fiber cell wall stretches the primary wall membrane, which exercises a restriction on expansion and in doing so loses the characteristic folds of the untreated fiber.

A surprising result of the studies of mercerized fibers is the revelation that their surface is not a great deal smoother than that of native fibers. Although mercerization effectively removes many of the gross concavities of the fiber surface, the submicroscopic roughnesses remain. This result must be attributed to the relatively large difference between the perimeters of the primary wall and the secondary thickening of the fiber, differences which are not overcome by the swelling and shrinkage of these fiber components during mercerization.

The new properties conferred by chemical modifica-

tion of the cellulose also influence the resultant surface of the derivative fiber. Modifications which are sensitive to the solvent system of the reaction employed lose discreteness of the fibrillate texture of the untreated fiber surface. Undoubtedly the swelling and solvation which cause this to happen would lead to fiber bonding if allowed to proceed.

Acknowledgment

The authors wish to express appreciation to members of the Cotton Chemical Processing, and Cotton Mechanical Processing Sections of this laboratory for supplying many of the samples examined and information concerning them. The assistance of Miss Blanche R. Porter in the preparation of certain samples is also gratefully acknowledged.

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Nomograph for Extending the Range of the Arealometer

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IN evaluating fine and immature cottons by means of instruments operating on the principle of resistance to air flow, occasional samples are found which have values below the scale of the instrument. An analysis of the operation of the Arealometer,[2] which employs this principle, has shown that there are relationships between air-flow resistance and specimen size which can be used to extend its range to fine cottons not presently measureable and, in addition, to determine surface properties over a range of compressions. Thus, greater flexibility in use is afforded than by the present test procedure which offers only two degrees of compression. The mathematical and physical basis for these relationships is developed in this paper and their application discussed.

In the basic article on the Arealometer, Hertel and Craven [1] developed the equation

$$(AL)^{\frac{1}{2}} = \left(\frac{\zeta}{k_o}\frac{1}{\mu m^2 \rho}\right)^{\frac{1}{2}} R^{\frac{1}{2}}\rho\alpha L - \left(\frac{\zeta}{k_o}\frac{1}{\mu m^2 \rho}\right)^{\frac{1}{2}} R^{\frac{1}{2}} m \quad (1)$$

where L is the length of sample plug, A is the specific area, and m the specimen mass. If the orientation factor ζ, the shape factor k_o, the density

[1] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U.S. Department of Agriculture.

[2] Mention of names of firms or trade products does not imply that they are recommended or endorsed by the U.S. Department of Agriculture over other firms or similar products not mentioned.

ρ, the cross-sectional area α, and the viscosity of air μ, are assumed constant for a given resistance to air flow R, the above equation may be written as

$$(AL)^{\frac{1}{2}} = km^{-\frac{1}{2}}(\rho\alpha L - m) \tag{2}$$

or further reduced to

$$\left(A^2L\frac{L}{m}\right)^{\frac{1}{2}} = k_1\frac{L}{m} - k_2 \tag{3}$$

Thus, the specific area A is related to the length of plug L and mass of specimen m provided the constants k_1 and k_2 can be evaluated.

The term L/m is left in this form because of its relationship to the void fraction, ϵ, which is the ratio of void volume to total volume.

$$\epsilon = \frac{V_t - V_c}{V_t} = 1 - \frac{4}{\pi d^2\rho}\left(\frac{m}{L}\right) \tag{4}$$

where V_t and V_c are the total volume and the volume occupied by the cotton fibers, respectively, d is the specimen chamber diameter, and the other terms are as defined under Equation 1. For the Arealometer with a fiber chamber of 0.319 in., Equation 4 becomes

$$\epsilon = 1 - 0.502\left(\frac{m}{L}\right) \tag{5}$$

where ϵ is the void fraction, and where L is expressed in inches and m in grams.

The evaluation of the constants in Equation 3 above may be performed by assuming the normal mass, 0.152 g., and measuring the plug length for two values of A on the low compression scale. This operation is facilitated by use of the table of A versus L which was furnished with the original model of the Arealometer [3] because it was calibrated in L rather than directly in A. In the older model, there was a "Medium" range which is identical with the "Low" range of the present instrument. Choosing two pairs of values for A and L from this table and substituting in two simultaneous equations from Equation 3 above, the constants are evaluated and the following equation results:

$$\left(A^2L\frac{L}{m}\right)^{\frac{1}{2}} = 31.04\frac{L}{m} - 15.20 \tag{6}$$

where A is in mm.^{2}/mm.^{3}, L is in inches, and m is in grams.

Equation 6 allows the computation of A for any length of sample plug when the mass is known. For example, if a mass 75% of normal, i.e., 0.114 g., is chosen the following equation results:

$$(A^2_{75\%}L^2)^{\frac{1}{2}} = 132.1L - 7.372 \tag{7}$$

where $A_{75\%}$ represents the specific area of the sample when 75% of the normal specimen mass is used.

In the following table a series of values for L were chosen, and $A_{75\%}$ was calculated from Equation 7 above. Also shown are values for A_d, which is the dial reading for the present model of the Arealometer and identical with $A_{100\%}$. The values of A_d were taken from the table of A versus L mentioned above.

L, in.	$A_{75\%}$ mm.^{2}/mm.^{3}	A_d, dial reading
0.500	899	634
0.400	767	528
0.300	611	407
0.200	416	253

The calculations entailed by Equation 6 are rather tedious. However, if $A_{75\%}$ is plotted against A_d, an approximately straight line relationship is found which over the usable range may be expressed as

$$A_{75\%} = 1.268A_d + 95 \tag{8a}$$

Since Equation 8a provides a simple relationship, similar equations have been computed for a range of specimen masses from 75 to 95%.

$$A_{80\%} = 1.203A_d + 69 \tag{8b}$$

$$A_{85\%} = 1.137A_d + 51 \tag{8c}$$

$$A_{90\%} = 1.088A_d + 31 \tag{8d}$$

$$A_{95\%} = 1.040A_d + 16 \tag{8e}$$

These relationships between A_x and the dial readings are plotted in Figure 1 for the various sample masses expressed as a percentage of normal mass. Figure 1 therefore provides a nomograph for converting the dial reading with specimen plugs of the various masses to the specific area, A.

Also plotted in Figure 1 are values for the void fraction, ϵ, ranging from 0.675–0.85 computed from Equation 5 and the table for converting length of specimen plug to specific area values. Thus, a further application of Figure 1 is to show the mass of specimen needed, for a given cotton, to give any desired void fraction. For example, cottons in the

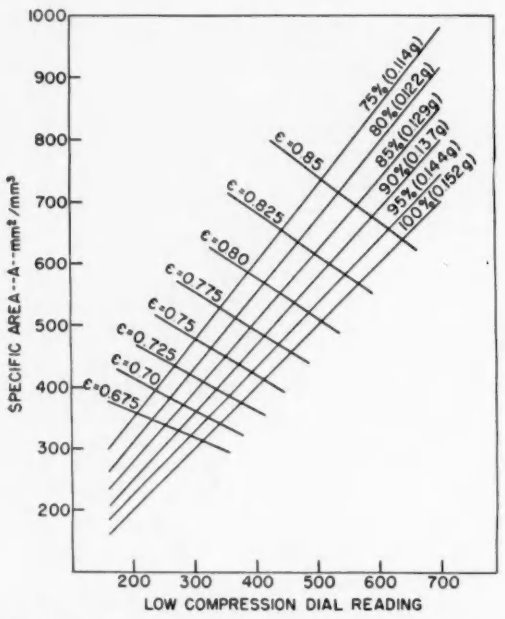

Fig. 1. Nomograph for converting Arealometer "Low Compression" dial readings to specific area for a range of specimen masses, and for indicating the void fraction, ϵ.

410 to 480 mm.^{2}/mm.^{3} range may all be tested at approximately the same void fraction, 0.75, by varying the mass of the specimen within the range given in the nomograph. This may provide a means for studying the effect of packing on the apparent specific area of the sample.

If desired, Figure 1 can be expanded to include sample masses greater than 100% of the normal, for there appears to be no practical or theoretical reason preventing such an expansion. Such an extension would be helpful in evaluating very coarse cottons, for examples, Peruvian Iquitos and coarse Indian cottons.

The question naturally arises as to the feasibility of similarly extending the scale of the Micronaire instrument. While theoretically possible, the practical difficulty lies in the empirical nature of the present curvilinear scale. Therefore, the only solution appears to be the empirical development of tables for converting the value read when using an altered specimen mass to the present Micronaire scale, or an extension of it.

The use of the 75% mass curve has been in use at this laboratory for a considerable time. The relationship has been confirmed by measuring a number of cottons in the high specific area region using specimens with both normal and 75% of normal mass. The degree of agreement has been good. More recently, the manufacturers of the Port-Ar (and Arealometer) have confirmed these findings and have recommended use of a 6-g. rather than an 8-g. (i.e., 75% of normal) specimen when testing fine cottons [2]. They also recognize the difficulties in making a similar conversion for their "Micronaire Curvilinear Scale."

Acknowledgments

Appreciation is expressed to Annie B. Cooper and Dorothy C. Legendre for work in confirming the relationships.

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The Resistance of Sized Yarns to Abrasion

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Abstract

The laboratory tests which are now in use for assessing the weavability of sized warp yarns are reviewed. Reference is made to the "dummy loom" type of test in which the elements of abrasion, flexure, and cyclic loading are combined. Some data are presented on the tensile properties of sized yarns before and after they have been subjected to sustained abrasion in the dummy loom. A study of these properties for different amounts of size pickup and different extents of abrasion has confirmed the hypothesis that the effect of this type of abrasion is to remove the size film at varying rates from different parts of the yarn. Some data are presented which suggest that the transient abrasive action of actual looms on sized warp yarns is similar in kind though much less severe in degree.

Introduction

The most reliable way of assessing the weavability of sized yarns is to carry out large scale weaving trials. The time and expense of such tests may be reduced by using properly designed experiments [2, 3, 4, 8, 14] which yield the maximum possible information. Use can also be made of special slashers [1] which permit sections of warp ends, processed in different ways, to be mounted side by side on the same weaver's beam. In spite of these short cuts, the assessment of weavability is still a troublesome affair in a production department. A certain amount of interest, therefore, centers on the question of laboratory tests for weavability [6]. The present paper deals with one aspect of this question, namely, the tensile behavior of yarns which have been abraded by healds and reeds.

Laboratory Tests for Weavability

There is plenty of experimental evidence [11] to show that tensile tests on sized warp yarns do not help in predicting breakage rates in weaving. This is not very surprising when the following facts are considered:

a. The mean and the coefficient of variation of tensile strength or elongation are insensitive to the presence of the very small number of very weak places which actually rupture during weaving unless an impracticably large number of tests is done. It is known [5, 7, 12] that the maximum tensile load and extension suffered by any yarn during a weaving cycle come to only a fourth or less of the mean tensile strength or extension.

b. During weaving, new weak places are created on account of abrasion by healds and reeds. These can be detected only by means of tensile tests on the abraded yarn.

c. Even strong ends may break when knots or imperfections are trapped by constrictions in the loom.

Reliable laboratory tests of weavability aim at subjecting the yarn to sustained flexure combined with abrasion against yarns [13, 15], or against metal surfaces [10], or against healds and reeds. This last type of abrasion has been secured by other workers in this laboratory on a simulated or "dummy" loom [9]. In the dummy loom, a sheet of stationary warp yarns is clamped in a fixed jaw at one end. The yarns are drawn through healds and reeds, just as in an ordinary loom. The other end of the sheet is guided round a pulley and clamped to a second free jaw, which can be weighted. The available loom movements are shedding and reed traverse. These movements are kept geometrically similar to actual weaving practice and take place at the same rate as in the weaving operation. However, the warp yarns are stationary and therefore receive sustained abrasion, while no weft yarns are inserted.

These tests measure the rate at which breakages occur as the abrasion proceeds. Such breakage rates have been found to be helpful in ranking yarns in order of warp breakage rates during actual weaving. It was therefore felt that some more useful information would emerge from the tests if the tensile prop-

erties of the survivors were studied after the test was over.

The Tensile Properties of Abraded Yarn

In order to establish the tensile properties of sized yarns after abrasion, two yarn sets having counts of 32 and 40 were selected. They were sized in a conventional manner in a mill and also in a laboratory slasher. The abrasion was conducted at the rate of 180 cycles/min. in each case on 100 sized ends in the dummy loom, under a static tension of 10 to 15 g. per thread, until 20 ends broke. The load-extension curves of the 80 survivors were plotted one by one on the Instron Tensile Tester. The testing conditions were $82 \pm 2°$ F. and $62 \pm 3\%$ R.H. Similar experiments were conducted with 100 ends of the corresponding unsized yarn. In this case, of course, a much smaller number of abrasion cycles on the dummy loom sufficed to produce 20 breaks. From the load-extension curves, the measured tensile strength and elongation were plotted in the form of frequency polygons. The results which were obtained for yarns sized in the laboratory are shown in Figure 1.

It will be clear that the effects of abrasion are as follows:

1. For unsized yarns, the frequency distribution of tensile strengths shifts bodily toward smaller mean strengths without much change in the shape of the curve.

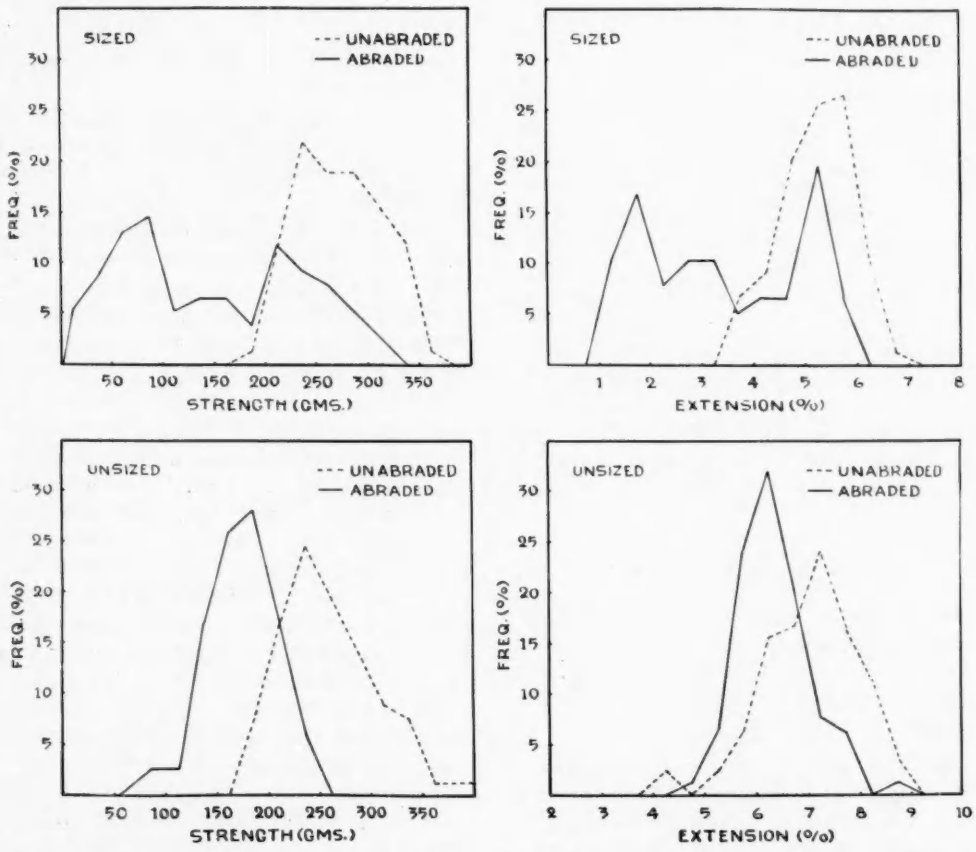

Fig. 1. Abrasion resistance of laboratory sized yarn. Sized and unsized yarns behave differently toward sustained abrasion on the dummy loom, as seen from the distributions of their single end strength and extension, before and after abrasion. The pronounced flattening of the distribution for sized yarns on abrasion indicates creation of new weak places in the process of abrasion. In all the cases, the distribution for strength is essentially similar to that for extension.

2. For sized yarns, the frequency distribution becomes bimodal in favor of a new population of considerably weaker yarns.

3. For both sized and unsized yarns, there is a close similarity between the distribution curves of strength and extension.

This experiment was repeated with the mill sized yarns, and the results were as shown in Figure 2.

While there is no pronounced bimodality of strength of the sized abraded yarns, there is still a considerable flattening of the distribution curve, indicating the creation of a new population of weak places. Repeated experiments have confirmed that this type of distribution generally results when sized yarns are subjected to prolonged abrasion in the dummy loom.

The Effect of Size Percentage on Abrasion Resistance

The next series of experiments was designed to find out how far the change in tensile properties on abrasion was dependent on the percentage of size (present on the yarn). Four sets of 100 ends, sized at 8.8, 7.9, 5.7, and 3.2% size, were prepared and each set abraded to 20,000 cycles on the dummy loom, producing 20, 19, and 25 breaks in the first three cases. In the last case (3.2% size) all the yarns broke before 20,000 cycles. The strength distribution curves of the survivors are shown in Figure 3.

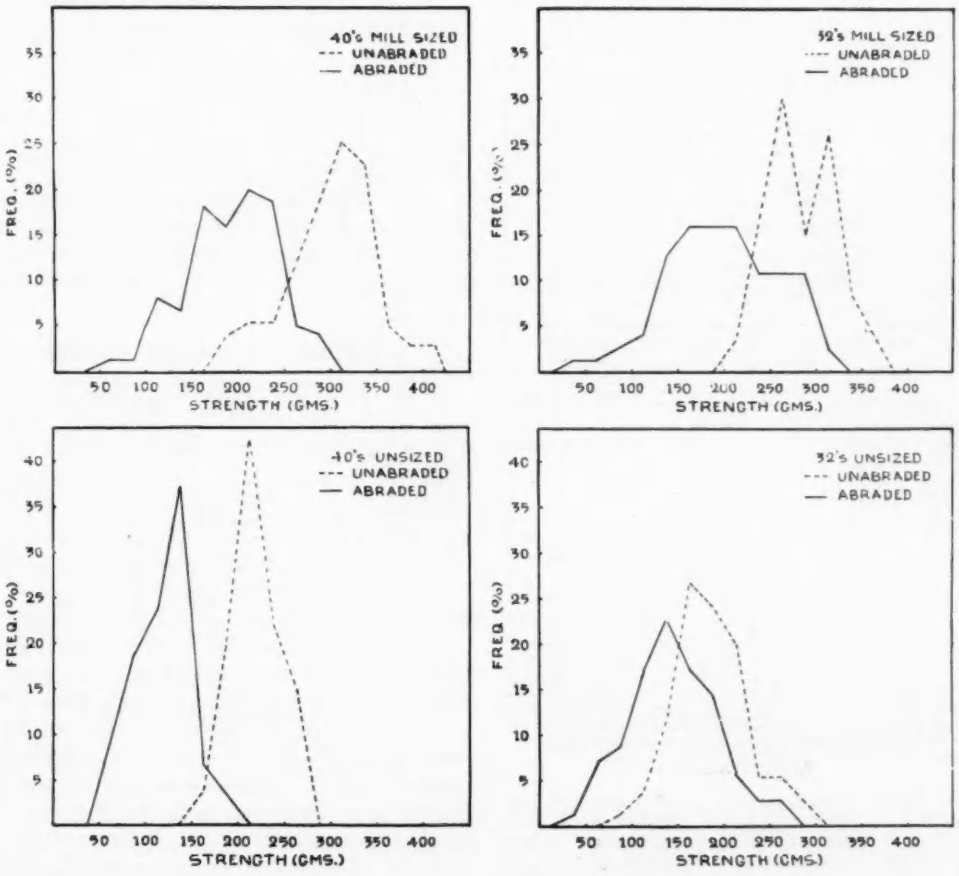

Fig. 2. Abrasion resistance of mill sized yarn. The same effect as in Figure 1 is shown in the case of two yarns of different count, sized in different ways in a mill.

The flattening effect becomes more pronounced as the size per cent is decreased. It is difficult to distinguish between the 8.8 and 7.9% curves in the matter of abrasion resistance. This agrees with the fact that the number of breaks is also much the same in both cases. Figure 3 also shows the tensile strength distribution curves of these same yarns before abrasion had taken place. The curves are bunched together and it is impossible to say which amount of size is better. Nevertheless, there are wide differences in the abrasion resistance of the yarns, as we have just seen.

The Effect of Increasing Abrasion

In the third series of experiments, the tensile strength distribution curves were plotted for increasing severities of abrasion. For this purpose, sets of 100 ends of yarn carrying 8.8% size were abraded at 4, 8, 10, 12, 14, and 16×10^3 cycles in the dummy loom. The breakages were counted and the tensile strengths of the survivors determined as before. Two loading arrangements were used in the abrasion experiments:

a. In the first case, the weighted clamp in which the yarn sheet was fixed carried a constant load. Thus the tension per thread increased from break to break.

b. In the second case, the weighted clamp carried an adjustable load. This load was manually decreased in the course of the test so that the tension per thread remained constant as breaks occurred.

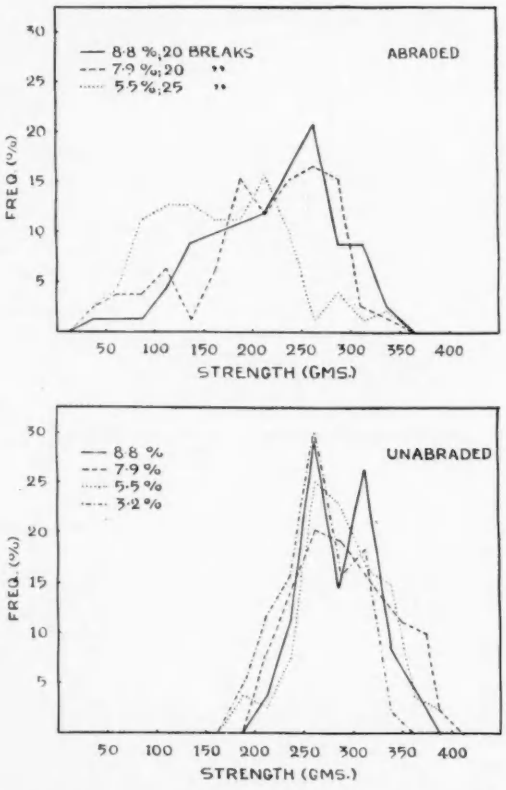

Fig. 3. Effect of size per cent on abrasion resistance. Yarns containing less size develop more weak places when subjected to the same amount of attrition in the dummy loom. But before abrasion has taken place, their tensile strength distributions are much the same. Note particularly the case of the 3.2% size, which gave no survivors after abrasion.

The strengths of the survivors are shown in the cumulative frequency distribution of Figure 4.

In this diagram, each ordinate represents the fraction of the survivors having a strength equal to or less than the corresponding abscissa. Taking any strength value, the effect of increasing abrasion is to make an increasing fraction of yarns weaker than this strength. Thus the curves steadily flatten out and shift to the left as the abrasion is increased. By inspecting these diagrams, it is possible to rank the yarns unambiguously in order of increasing abrasion.

Comparisons with Weavability

It was mentioned at the outset that the breakage rates obtained with yarns on a dummy loom showed a good correlation with the breakage rates obtained in weavability tests. It was therefore thought worthwhile to see if the results of weavability tests were also related in any way to the tensile strength distribution of yarns which survived abrasion in the dummy loom. For this purpose, four beams were available which had been sized in four different ways from the same grey lot. Weavability tests had previously been conducted on these four beams in order to determine the efficacy of four china clays (E, K, G, H) each of which was tried out individually in a sizing recipe which was otherwise the same. One hundred ends in each set were selected and given 20,000 cycles of abrasion in the dummy loom. The strengths of the survivors were determined and plotted in the form of cumulative frequency distributions. These are shown in Figure 5, which also shows the warp breakage rates per 10^4 picks observed with

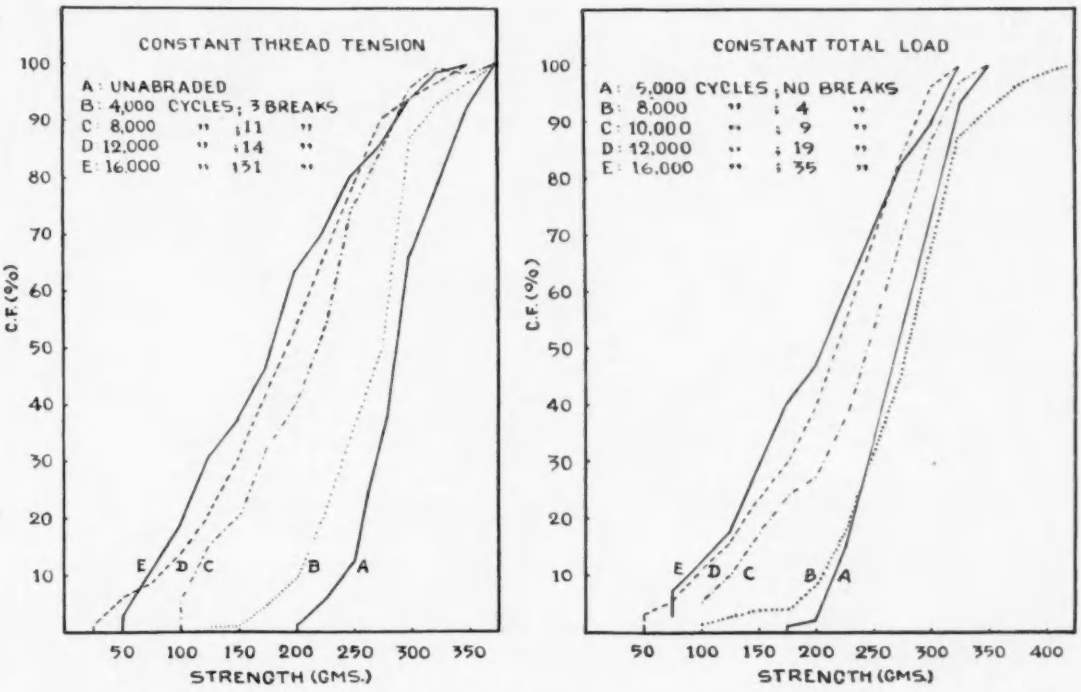
CONSTANT THREAD TENSION
A: UNABRADED
B: 4,000 CYCLES; 3 BREAKS
C: 8,000 " ; 11 "
D: 12,000 " ; 14 "
E: 16,000 " ; 31 "
C.F.(%)
STRENGTH (GMS.)
CONSTANT TOTAL LOAD
A: 5,000 CYCLES; NO BREAKS
B: 8,000 " ; 4 "
C: 10,000 " ; 9 "
D: 12,000 " ; 19 "
E: 16,000 " ; 35 "
C.F.(%)
STRENGTH (GMS.)

Fig. 4. Effect of increasing abrasion on tensile strength. In these curves, the ordinates give the fraction of yarns having lower strengths than the value on the corresponding abscissae. This fraction steadily increases with increasing attrition.

these yarns in the loomshed. The breakage rates have been corrected for breaks due to crossed ends.

It will be clear that when the extreme left tails of these distributions are considered, the yarns are ranked according to degree of flattening in the same order in which they are ranked according to weaving breakage rates. On the other hand, when the percentage of yarns having strengths below 150 g., is considered, the orders of ranking change very much. It is on account of this fact that the cumulative frequency distribution curves have been drawn only up to 200 g.

Discussion of Results

All the experiments described so far have a common feature in that when sized yarns are abraded, there is a progressive creation of very weak places. It is as though the size film affords a varying amount of protection to the yarn structure inside. In some places, the size film is rubbed off early in the course of abrasion and the bare yarn surface is exposed to

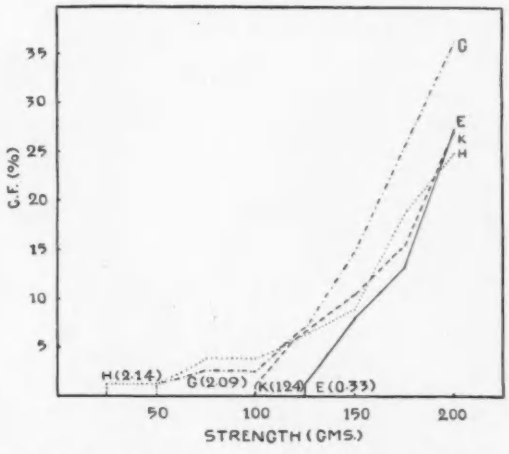

Fig. 5. Comparison of different sizes. The yarns *E*, *K*, *G*, *H* were sized in four different ways from the same grey lot. They were evaluated by drawing the cumulative frequency curves of strength after attrition in the dummy loom. The position of the extreme left tail of these curves rank the yarn in the same order as the relative warp breakage rates, which are indicated in brackets in the chart.

the action of the healds or reed. This ruptures the yarn at the point of exposure or leaves it in a frayed, untwisted, and weak condition. There are on the other hand, also places where the size film adheres tenaciously, so that the yarn is fully protected or only slightly weakened during the course of abrasion. This is a ready and simple explanation for the presence of very weak ends as well as the quite strong ends noticed after abrasion. When the size film is very irregular, and the tendency for local flaking becomes pronounced, the superposition of strong and weak places becomes clearly marked as two distinct modes in the bimodal distribution of Figure 1. Likewise, when the average size per cent is low, the chance of more places being inadequately protected by the size film becomes greater. This explains the results obtained with decreasing size per cent. In the same way, it is easy to understand how increasing abrasion takes off all the size from more and more ends and gives rise to the series of curves shown in Figure 4.

The results obtained with unsized yarn are totally different. Since there is no protective size film in this case, the abrasive action is much more pronounced and a few cycles on the dummy loom may make all the difference between slight attrition and total rupture.

These experiments suggest that from the point of view of resistance to prolonged and severe abrasion by healds and reeds, a good sizing should show the following properties as compared with a bad sizing:

a. Less yarn breaks for a given number of cycles on the dummy loom.

b. Less area in the "left tail" of the frequency distribution curve of strength or extension of the survivors.

c. Higher limiting minimum strength and less pronounced "trailing" of the left tail of the strength distribution curve.

The question now arises as to whether these criteria can be applied to judge the weavability of sized yarns. The last series of experiments shows that they can be so applied if we limit ourselves to very low strengths in defining the "left tail." However, more work has to be done before settling the point and assigning an unambiguous meaning to the "left tail." In doing this work, the following points should be borne in mind:

a. Several hundred ends should be abraded at a time rather than just 100.

b. The level of abrasion should be milder than what has been used until now but still severe enough to cause measurable results among the several hundred ends.

c. Tensile testing of the survivors should be conducted in a much quicker way than by repeated single-end testing.

d. The experiments should be easily duplicable in order to furnish figures for reproducibility.

A machine which incorporates all these features is being designed and built in ATIRA. It is hoped that this instrument will furnish an answer to one (and probably the most important) aspect of the question of laboratory tests for weavability, namely, the resistance of sized yarns to abrasion.

Acknowledgment

We are grateful to the Council of Administration of ATIRA for giving us permission to publish this paper. We would also like to express our thanks to Messrs C. C. Shah, R. V. Soneji and M. S. Thadani who performed the experimental work on sizing and dummy loom abrasion.

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Some Thermal Properties of Fabrics[1]

Part I: The Effect of Fiber Arrangement

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Abstract

Examination of the thermal conductivity of fabrics in terms of the fiber conductance and fabric density shows that fabric insulation is determined by fiber arrangement as well as by fabric thickness. Fiber arrangement is interpreted in terms of fibers lying parallel and perpendicular to the fabric surface, and it is shown that fabrics differ appreciably in this respect. For the smooth surfaced fabrics of cotton, nylon, and Orlon studied, the increase in specific conductivity with applied pressure can be attributed mainly to an increase in fabric density as there is very little change in fiber arrangement in such fabrics. For fuzzy surfaced fabrics of wool or blends containing wool, the change in density with applied pressure is counterbalanced by changes in the fiber arrangement. At low pressures a substantial portion of the fibers lie perpendicular to the fabric surface but are bent over as the pressure is increased. At high pressures the fibers lie mainly parallel to the surface, but the fabric conducts less effectively because of the air layers between each fiber along the conducting path. Accordingly the specific conductivity of wool and wool-type fabrics is relatively insensitive to changes in applied pressure and this appears to be a direct result of the random arrangement of the fibers in these fabrics.

Introduction

It is generally agreed that the insulating characteristics of fabrics are related to the air entrapped by and surrounding the fibers. Thus efforts to achieve improved insulation have been directed toward decreasing the bulk density, consistent with other functional requirements, with the idea of increasing the concentration of the air component.

Recent studies of blended serges [4] have suggested that the arrangement of fibers in the yarn can effect a number of fabric properties, e.g., water transport, handle, coolness-to-touch. Work carried out on the thermal transmission of these blends indicated that the insulation properties were also influenced by fiber arrangement in the fabric. The purpose of this paper is to describe the results of measurements of thermal conductance of several fabric types over a range of pressures and to analyze the results in terms of fiber arrangement.

Heat Flow in Mixtures of Fiber and Air

The conductance of mixtures in terms of series and parallel elements occupied the attention of many physicists toward the latter end of the 19th century. Baxter [1] has reviewed and discussed in some detail much of the early work so that this will not be reiterated here. Finck [5] studied fibrous insulation of various kinds and concerned himself specifically with the effect of fiber arrangement. He demonstrated in a series of experiments with flax fibers, glass wool, and hair felt, among others, that when the fibers were arranged parallel to the direction of heat flow the thermal conductivity was two- to threefold greater than when assemblies of equal bulk density contained fibers arranged perpendicular to the direction of heat flow. As in the earlier studies, Finck interpreted his results in terms of series and parallel conducting elements in each assembly.

For a fiber-air assembly in which the conducting elements are arranged parallel to the heat flow, the specific thermal conductivity is given by

$$k = v_f k_f + v_a k_a \qquad (1)$$

in which k_f and k_a are the specific thermal conductivities of fiber and air, respectively, and v_f and v_a are the corresponding volume fractions of each component ($v_f + v_a = 1$). In such an arrangement, the high conductivity of the fiber (relative to that of air) contributes its maximum effect on the over-all con-

[1] This work constitutes a part of the Army Quartermaster program of research on warmth and comfort of military fabrics performed under Contract QM 564.

ductivity of the assembly. For a fabric, this type of arrangement corresponds to one with the fibers normal to the plane of the fabric as in a carpet or pile construction.

For a fiber-air assembly in which the conducting elements are arranged perpendicular to the direction of heat flow, the specific thermal conductivity is given by

$$k = k_f k_a / (v_a k_f + v_f k_a) \quad (2)$$

In such a series arrangement of conducting elements the high conductivity of the fiber material contributes a minimum amount to the over-all thermal conductivity of the assembly because of the air layer between each fiber element. For a fabric this corresponds to having all the fibers parallel to the plane of the fabric surface but each separated from its neighbor by a column of air.

In general, then, the specific conductivity of a fiber-air assembly is given by

$$k = x(v_f k_f + v_a k_a) + y k_f k_a / (v_a k_f + v_f k_a) \quad (3)$$

in which x and y are the effective fractions of fibers parallel and perpendicular to the direction of heat flow, respectively, and $x + y = 1$.

Using the thermal data of Finck [5] on fiber assemblies of flax, glass, and hair, values of x and y were calculated using Equation 3 and specific conductivity figures for the fibers from the literature. For assemblies in which the flax, glass, and hair fibers were arranged parallel to the direction of heat flow, the calculated values of x were 0.91, 0.81, and 0.86, respectively. For fibers arranged perpendicular to the direction of heat flow, the calculated values of y were 0.98, 0.96, and 1.00, respectively. Such excellent agreement between theory and experiment indicated that Equation 3 might be very suitable for assessing differences in fiber arrangement in blended fabrics.

The Measurement of Thermal Conduction

Thermal conductivities of the fabrics were measured using a two-plate thermal apparatus like that of Fitch [6]. Under the classification given by Morris [12], this involved the use of the "disc method" in which the rate of flow of heat between two metal plates at different temperatures is measured. The apparatus used in this work was modified for measurements of fabric conductance over a range of applied pressures. To accomplish this end, plate separation was controlled by a conventional lathe tool feed and measured with a dead weight thickness gauge. By a suitable choice of thermostatic equipment, it was possible to hold one of the plates of the instrument either above or below room temperature. For purposes of simulating freezing conditions this plate could be held as low as $-10°$ C.

Fiber Arrangement and Fabric Type

Thermal measurements on several conditioned fabrics varying in fiber material and construction were made at plate separations corresponding to fabric thicknesses at pressures from 0.002 to 1.0 lb./in.^{2}. The specific thermal conductivities of these fabrics relative to air, measured under the same conditions, are summarized in Table I. Using Equation 3 and the relative specific thermal conductivity of the fiber materials (Table II), the effective fraction of fibers normal and parallel to each fabric surface were calculated and these values are also given in Table I. The volume fraction of fibers, v_f, was estimated from the ratio of the bulk fabric density to the mean density of the fiber materials in each fabric.

The results for the wool fabrics show that as the thermal measurements are made at increasingly higher pressures, the specific thermal conductivity of each fabric does not alter very greatly. However the thickness does decrease, thus effecting an increase in bulk density and in the volume fraction of fibers between the test plates. The calculated fractions x and y indicate that the effective fraction of fibers parallel to the fabric surface increases as pressure increases. This means that the vertical pile-like fibers are bent over by compression such that an increasingly effective insulating arrangement of fibers and air is produced at higher pressures. Thus, for the wool fabrics examined, the increase in fabric density is compensated for by a decrease in fraction of fibers normal to the fabric surface, so that little change in fabric conductance is observed with pressure. It should also be noted that at very low pressures for the wool fabrics, the effective fraction of fibers normal to the surface is high, i.e. they are fuzzy or "wool-like." What prevents them from being highly conductive is the low bulk density.

In contrast to this behavior, the cotton, nylon, and Orlon fabrics measured exhibit a type of fiber arrangement in which the effective fraction of fibers arranged parallel to the fabric surface is appreciable even at low pressures. This is consistent with ex-

TABLE I. Fraction of Fibers Arranged Normal and Parallel to the Plane of the Fabric Surface

Sample No.	Fabric description	Fabric thickness,* mils	Fabric density, g./cm.³	Volume fraction fiber, v_f	Relative specific thermal conductivity, air = 1	Effective fraction of fibers in fabric†	
						Normal to surface, x	Parallel to surface, y
A	Smooth wool serge	86 (.002)	0.21	0.16	1.65	0.51	0.49
		66 (0.01)	0.27	0.20	1.79	0.49	0.51
		46 (0.1)	0.38	0.29	1.79	0.27	0.73
		35 (1.0)	0.48	0.36	1.82	0.15	0.85
12	Wool shirting	155 (0.01)	0.13	0.10	1.65	0.97	0.03
		131 (0.1)	0.15	0.11	1.74	0.95	0.05
		94 (1.0)	0.20	0.15	1.77	0.88	0.12
D	Cotton sateen	44 (0.01)	0.31	0.21	1.94	0.13	0.87
		32 (0.1)	0.42	0.28	2.21	0.12	0.88
		24 (1.0)	0.57	0.38	2.54	0.10	0.90
N	Nylon serge	67 (.002)	0.24	0.21	1.65	0.25	0.75
		43 (0.1)	0.38	0.33	1.84	0.16	0.84
		37 (1.0)	0.44	0.39	2.27	0.24	0.76
17	Wool serge	103 (.002)	0.17	0.13	1.72	0.78	0.22
		41 (1.0)	0.40	0.30	1.65	0.22	0.78
28	50 Orlon, 50 wool serge	74 (.002)	0.20	0.16	1.65	0.43	0.57
		34 (1.0)	0.43	0.34	1.79	0.14	0.86
20	Orlon serge	38 (.002)	0.33	0.28	1.66	0.15	0.85
		28 (1.0)	0.46	0.39	1.94	0.13	0.87

* Distance between plates of thermal apparatus. Numbers in parentheses are estimates of pressure (lb./in.²) exerted on fabric.

† Using Equation 3 and the relative specific thermal conductivities for different fibers of Table II.

TABLE II. Thermal Conductivities and Densities of Fiber Materials and Air

Material	Density,* g./cm.³	Specific conductivity, cal. in./m.² sec. C.°	Relative conductivity, air = 1	Conductivity reference
Air	—	0.23	1	[1, 7, 10, 13]
Wool	1.32	2.10	9	[1, 14, 15]
Nylon	1.14	2.4	10	[8]
Orlon	1.17	—†	—†	
Viscose	1.52	2.5	11	[1, 8]
Cotton	1.50	6.1	27	[1, 15]

* From du Pont Technical Manuals for nylon, Orlon, Dacron Physical Chemical Properties Sheet 2–5.01, July 1952.

† In the absence of directly measured values for Orlon, the specific thermal conductivity of nylon was used in the calculations for Table I.

perimental evidence on the nature of the surfaces of these fabrics [9] and with the visual and tactile impressions of these materials. As was pointed out in development of the fabric conductivity equations, this type of fiber arrangement is efficient for insulation purposes because of the alternate layers of fiber and air. Hence at low pressures, where the bulk density is low, the specific thermal conductivities of wool, cotton, and all-synthetic fabrics are similar despite the lower bulk density of the wool fabrics. When the pressure is increased, however, there is no opportunity for increasing the efficiency of arrangement further, i.e. there are few surface fibers to compress. The net effect is only to increase the bulk density to high values where high fiber conductance plays an increasingly important part in increasing the fabric conductivity.

It is of interest that the calculated values of x, the effective fraction of fibers normal to the plane of the fabric, seem to relate to the general visual and tactile impressions of the fabrics (Table I). Thus sample *A* is a smooth wool serge compared with sample 17, a relatively hairy fabric, and the napped wool spun shirting, sample 12, is clearly fuzzier than either. The series of fabrics of comparable construction, 17,

28, and 20, has been shown [4] to exhibit decreasing surface hairiness with decreasing wool content by means of two very different techniques [3, 9]; at similar pressures the proportion of fibers normal to the surface (Table I) decrease in the same sense.

Conclusions

This type of analysis suggests that the thermal resistance of fabrics is not only proportional to their thickness but also changes with applied pressure. For fuzzy fabrics of wool-like character, the decrease in resistance with pressure is counterbalanced by a change of fiber arrangement in the fabric so that the over-all resistance remains essentially unaltered even at high pressures. For smooth fabrics of cotton and the synthetic fibers, decrease in thermal resistance with pressure is appreciable as fiber arrangement undergoes only a minor change. The differences between the various equations which have been developed for relating fabric thickness to thermal resistance [2, 12] lie mainly in the pressure that was applied during the thermal measurements and indeed the expressions applicable at low pressures show good agreement for many types of fabrics, as would be expected from the present analysis.

Some emphasis should be placed on the fact that the conditions used in deriving the conductivity Equation 3 involved fiber arrangement models which were definitely ideal compared with the structure of real fabrics. However, the analysis yields a picture of fiber arrangement which appears to be consistent with other fabric characteristics. The conclusions which have been reached can be generalized as follows:

1. Fabric conductance is dependent on bulk density particularly as the proportion of fibers normal to the fabric surface increases.

2. At a given bulk density, fabric conductance is not very sensitive to fiber conductance when the fibers are arranged parallel to the fabric surface, and conductance must occur through alternate layers of fiber and air. As the number of normal fibers increases, the fabric conductivity becomes somewhat more dependent on fiber conductivity, especially at higher bulk densities.

3. Fabric conductivity is more sensitive to fiber arrangement as the fiber conductivity and bulk density are increased.

4. At a given bulk density, fabrics made of higher density fibers are better insulators since the volume fraction of fibers is determined by the ratio of bulk density to fiber density.

It is clear that better thermal resistance may be achieved, in terms of this analysis, by the use of (1) low conductance fibers, (2) a mechanically stable arrangement of fibers parallel to the surface, and (3) low bulk density. For conventional fabrics in single layers, requirements (2) and (3) seem to be antithetical. It is interesting that insulating batts or fiber boards are formed in a manner to conform to these requirements. Quilted fabrics or multilayer assemblies could well be visualized as conforming to the most desirable conditions for good clothing insulation, and this advantage has been demonstrated in recent thermal studies [11]. In these situations then, differences in fiber conductance may be of only minor importance if effective over-all fiber arrangement and low bulk density can be achieved. This fact is well illustrated in the thermal conductance measurements of Rees [13] on fleece fabrics of different fiber materials.

Acknowledgment

Appreciation is expressed to Dr. Alan H. Woodcock of the Environmental Protection Division of the Quartermaster Laboratories for helpful discussion of the manuscript as well as to Virginia A. Moran, Martha M. Kaessinger, and Barbara S. Watson who carried out the thermal measurements of this work. The authors are also indebted to Constantin J. Monego and Louis I. Weiner of the Textile Clothing and Footwear Division of the Natick Quartermaster Center for close cooperation throughout the course of this work.

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The Interference Microscope in Fiber Research[1]

Part I: Initial Studies on Rayon, Cotton, Wool, and Synthetic Fibers

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Abstract

Application of the Baker interference microscope in fiber research is described. It was found possible to reveal and distinguish fine details of the fiber structure and to recognize even minute differences in refractive index by color contrast. It is furthermore possible to determine quantitatively the refractive index of these details. The theoretical background is considered and the technique employed described. Typical examples with different fibers are shown.

I. Introduction

The Baker interference microscope used in this study is a recent development, patented in 1950 [20]. It has been employed successfully in biological research, especially in cytological studies [1, 19]. There appears to be a dearth of information in the literature with regard to its use in the study of textile fibers. The only reference found thus far is a brief reference by Joshi and Preston [11]. These authors did not report, however, any results similar to those described in the present paper. The Baker instrument was selected for the work described because of certain important advantages in its operation.

This article is intended to furnish background information concerning the instrument and its possibilities for revealing and analyzing the fine structure of textile fibers. Some results of the initial studies by the author are shown and further work is in progress on a variety of fibers.

Use of the interference microscope will yield rapid and precise results when the correct techniques of sample preparation and manipulation of the instrument and its attachments are followed. Some of the precautions to be observed and techniques developed during the investigation will be described briefly.

Interference Microscopes

The principle of the interference microscope is similar to that of the polarizing and phase microscope, in that in all three types of microscopes inter-

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ference of light is used for obtaining information beyond that given in ordinary microscopy. There are basic differences from the polarizing and phase microscopes, as will be seen in the following discussion.

In the polarizing microscope, interference occurs between light rays which follow the same path in the object, but vibrate in two mutually perpendicular planes, into which the object separates the light. Thus information can be obtained on the refractive indices in these two directions (and the birefringence) of the object.

In the phase microscope, interference occurs between the direct and diffracted rays, originating from the same object detail. A phase difference is artificially added between these two beams by means of a phase plate placed in the microscope. Thus, local differences in refractive index of the object are converted into differences in contrast in the image.

In the Baker interference microscope, interference occurs between components passing through the object and those passing through the mounting medium or the surround (hereafter termed "the medium"). In contrast to the instruments described above, the interfering beams do not originate from the same object point, but from different points of object and medium. Thus, the refractive index of the specimen detail is compared with that of the medium.

It is believed that the excellent results obtained in this study could probably not have been obtained with other types of interference microscopes, which give either basically different results or are less appropriate for the investigation of fibers. Many schemes for obtaining interference contrast are not only extremely complicated but also impractical for this purpose. The three other types of interference microscopes which are best known are the following.

In the multiple beam interference microscope, first designed by Tolansky [21], the interference occurs between rays directly passing through the object and rays that have been reflected backward and forward several times between a film of high reflectivity and low absorption, either on the surface of the object (Tolansky) and the slide, or, in the microscopes by Frederikse [5] and Merton [14, 15], between the semi-reflecting surfaces of the slide and the cover slip. This principle is entirely different from that of the Baker microscope used in this study. The phenomena, results and their explanation also differ.

The principle of the Dyson interference microscope [2, 3] is about intermediary between that of the multiple beam microscope and the "round the square" and double focus systems, to which the Baker microscope belongs. The beam is split at a semi-reflecting surface closely below the object and just as in the previous type, the two beams are reunited before entering the objective, after several reflections at a few semi and totally reflecting surfaces. During these many reflections, a considerable loss of intensity occurs. A great disadvantage is that a separation of the two beams (in their passage through object and medium) is not so complete as in the Baker system. For this reason, the tolerable size of the object is very limited. The mechanism for adjustment of this rather complicated microscope is, moreover, delicate.

The oldest type of interference microscope is due to Linnik [13] and has been developed on a commercial basis by Zeisz [12]. One of the interfering beams is reflected on an optical flat and the other on the object surface or on a mirror, on which the object is placed, thus introducing a path difference.

Of the various existing types of interference microscopes, the Baker interference microscope is probably the best practical solution, in view of the following features. A complete separation of the pathways of the interfering beams through object and medium is achieved here (especially in the shearing system which is superior in this respect to the double focus system which can be used alternately with this microscope). No delicate mechanism for adjustment is used. The phase and intensity of the beams can be varied in a simple way and relatively large specimens can be studied without destroying the image.

II. Principle of the Baker Interference Microscope

The general principle of the A. O. Baker microscope is illustrated in Figure 1. A beam of light is separated by a beam divider and the separated beams A_m, B_m and A_m^1, B_s pass through different parts of object space. Rays passing through equal optical path, A_m and A_m^1 interfere to give rise to constructive interference. Rays passing through different optical path, B_m and B_s may give rise to destructive interference. Under the conditions of Figure 1, destructive interference will result since the specimen in the illustration has introduced a phase difference half a wave length in advance of the reference ray.

Figure 2 illustrates schematically the construction of the microscope. The microscope uses a polarizing

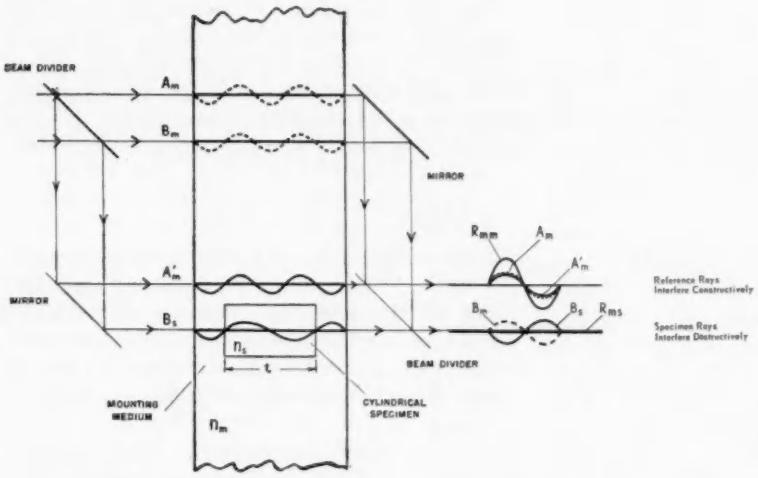


Fig. 1. Principle of the interference of light in interferometer and in the A. O. Baker interference microscope. (Courtesy of American Optical Company.)

beam splitter, consisting of a doubly refractive crystal. A similar crystal recombines the beams later on to form the image. (A ray of light incident on a crystal, such as calcite, will in general, emerge in two separate rays, the ordinary and extraordinary rays which are polarized in mutually perpendicular planes.) Lenses of this doubly refractive material provide an axial separation of the two beams. The beam splitting lens is a component of the condenser and the beam recombining lens a component of the objective. The light enters the microscope through a polarizer inclined at 45° to the optic axis of the beam splitter. The condenser brings one beam at a focus in the object plane and this beam traverses the specimen detail. The other beam is focused at the side of the object in the shearing system, or underneath the object in the double focus system and passes through the medium at the side of the specimen or spreads all around it. The separate beams are recombined by the birefringent component in the objective, so that they follow the same path and can interfere.

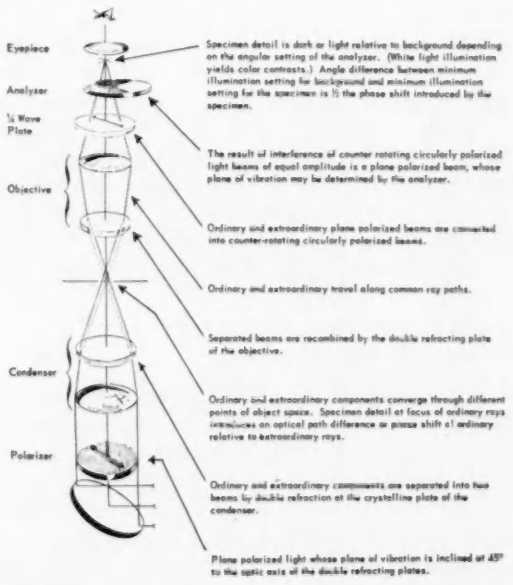

Eyepiece
Analyzer
¼ Wave Plate
Objective
Condenser
Polarizer
Specimen detail is dark or light relative to background depending on the angular setting of the analyzer. (White light illumination yields color contrasts.) Angle difference between minimum illumination setting for background and minimum illumination setting for the specimen is ½ the phase shift introduced by the specimen.
The result of interference of counter rotating circularly polarized light beams of equal amplitude is a plane polarized beam, whose plane of vibration may be determined by the analyzer.
Ordinary and extraordinary plane polarized beams are converted into counter-rotating circularly polarized beams.
Ordinary and extraordinary travel along common ray paths.
Separated beams are recombined by the double refracting plate of the objective.
Ordinary and extraordinary components converge through different points of object space. Specimen detail at focus of ordinary rays introduces an optical path difference or phase shift of ordinary relative to extraordinary rays.
Ordinary and extraordinary components are separated into two beams by double refraction at the crystalline plate of the condenser.
Plane polarized light whose plane of vibration is inclined at 45° to the optic axis of the double refracting plates.

Fig. 2. Optical system of A. O. Baker interference microscope. (Courtesy of American Optical Company.)

If the optical path through the specimen detail differs from that through the medium, the beams will have different phase. When the two rays are recombined to form the image, their interference will result in differences in contrast. If polychromatic light is used, some wave lengths will interfere destructively and others constructively, so that interference colors will result, just as in polarizing microscopy. Regions of different refractive index (optical path) will thus exhibit different colors. In this way, details of different refractive index can be clearly distinguished. Their refractive index can also be quantitatively determined, as will be described below.

III. Methods

Quantitative Measurement of Phase Difference

The phase difference can be measured by any of the methods customary in polarizing microscopy [8].

In the Baker microscope this is done with the use of a quarter wave plate inclined at 45° to the vibration directions of the two beams and a rotating goniometer analyzer [3]. The quarter wave plate converts the mutually perpendicular vibrations of the two component rays into corresponding circular vibrations which have opposite direction and equal amplitude. Interference of these vibrations results in a plane polarized vibration. It can be shown by a simple vector diagram that the angular inclination (with regard to the equator) of this resultant vibration equals half the phase difference between the two rays.

It is thus possible to determine quantitatively the refractive index of the object detail by the setting of the analyzer. According to the manufacturers of the microscope, the accuracy of the measurement of phase would be 1/120 or even 1/300th of a wave length. Further refinements of the instrument are said to be under development which will permit of still greater accuracy.

The measurements are made by reading the goniometer analyzer at minimum illumination setting for the medium and at minimum illumination setting for object detail. Monochromatic mercury green was used for this purpose (a Bausch and Lomb interference filter in combination with a mercury vapor lamp). The phase difference equals twice the angle between these two settings.

If the phase difference is more than 360 degrees and if a gradual change of the optical path from detail to medium or medium exists, a number of interference fringes will be visible in the object. The integral phase difference in wave lengths corresponds to the number of these fringes.

The refractive index can be calculated in the following way. If the refractive index of the medium is n_m and of the specimen n_s, and the thickness of the specimen is t, then the phase difference φ in degrees equals

$$\varphi = \frac{360}{\lambda} [(nt)_m - (nt)_s] \qquad (1)$$

Therefore, $n_m - n_s = \varphi\lambda/360\ t$, so that n_s can be calculated if n_m and the thickness t are known. Determinations of refractive index, of course, should be carried out with monochromatic light.

It is inversely possible to calculate accurately the thickness of a certain detail when the refractive indices of medium and specimen are known, using the same formula.

For obtaining satisfactory results, the proper setting of the microscope and adaption of the sample is necessary. This requires certain technical adjustments of the instrument and certain special requirements for the preparation of the specimen, of which the following are particularily important.

Illumination

Köhler illumination is prescribed for illumination. The best light source is either a mercury vapor or a ribbon filament lamp. The use of a spiral filament lamp is undesirable since in Köhler illumination, an image of the filament would be formed in the focal plane. This is undesirable in connection with the following.

The light entering the objective should originate from only one of the many interference fringes which are originally visible at the back focal plane of the objective. This is achieved by means of two substage screws by which the condenser can be tilted. In this way, only one fringe is brought in the center of the microscope and spread out, so that the back focal plane is completely and uniformly filled by it. This adjustment requires some practice but is essential for satisfactory results.

Size of Object Area

The area passed by the reference beam in the shearing system, for the 40 × objective, is a laterally displaced region of the field located at about 160 microns from the optical axis. The best measurements are obtained if this area does not overlap to a great extent with the object area. In the double focus system, the reference area surrounds the object and the separation of the two areas is much less perfect, when larger objects like fibers are studied.

Physical Conditions for Obtaining Color Contrast with Fiber Cross Sections

For the study of details of different refractive index by interference colors, it does not suffice that the above instrument setting is correct and that white polychromatic light is used. In the course of the present investigation it was found necessary to develop a few special techniques. The following theoretical discussion based on the findings in the course of the experimental work reported in the following part of this paper will be found to be helpful.

Clear, distinct interference colors were found to occur only if the section had a definite thickness in combination with a mountant of the proper refractive index. Colors of a clear contrast, therefore, result only if the object causes a path difference which lies within a limited range, namely above about 215 m. microns ($\frac{1}{4}$ wave length) and under about 2000 m. microns (about 3 wave lengths). Under 215 m. microns, the color varies between grey and first order white, and above the seventh order, the color goes back again to almost first order white. The path difference is determined (see Equation 1) by the thickness of the section and the difference of refractive indices of object and mountant.

The following calculations illustrate this. If a 6-micron thick cross section of rayon ($n = 1.53$) is mounted in ethyl salicylate ($n = 1.523$), the average path difference in water will be $(1.530 - 1.523) \times 6000 = 42$ mμ (which corresponds to a phase difference for $\lambda = 540$ mμ of about 28°). This difference will give rise only to interference colors of a greyish hue (see Table I). If the section is mounted in water ($n = 1.333$), the path difference will be 1182 mμ which will cause interference colors of the second order which have brilliant contrast. If the section is 20 microns thick, and mounted in water, the path difference is 3940 mμ), causing colors of the seventh order, in which no clear contrast exists.

The color photomicrographs, Plate I and IV, illustrate this for 6-microns thick viscose rayon sections mounted in liquids of different refractive index, namely, water (1.333) and glycerol (1.460). It is seen that a good color contrast is obtained only in water, and that in glycerol a decreased contrast and only a uniform color hue results.

IV. Results and Discussion

Observations with Fibers in Longitudinal View

If a cylindrical fiber, for instance a nylon or Dacron [3] filament, is mounted in water, and examined under the Baker microscope, a series of interference fringes is observed along the longitudinal direction of the fiber (Figure 3). This is a result of the increasing thickness of the fiber toward its center which causes an increasing path difference with the medium.

The fringes are most distinct when monochromatic light is used. The observations in this paper were made using a green Bausch and Lomb interference filter for a wave length of 546 m. microns. In the same liquid, the nylon filament shows a smaller number of fringes than the Dacron filament of the same diameter, because of the higher refractive index of the latter fiber. The number of interference fringes decreases when the difference in refractive index between mounting liquid and fiber decreases (Figure 3), until the two indices are equal or nearly so, in which case no more fringes are present (for Dacron

[3] Du Pont polyester fiber.

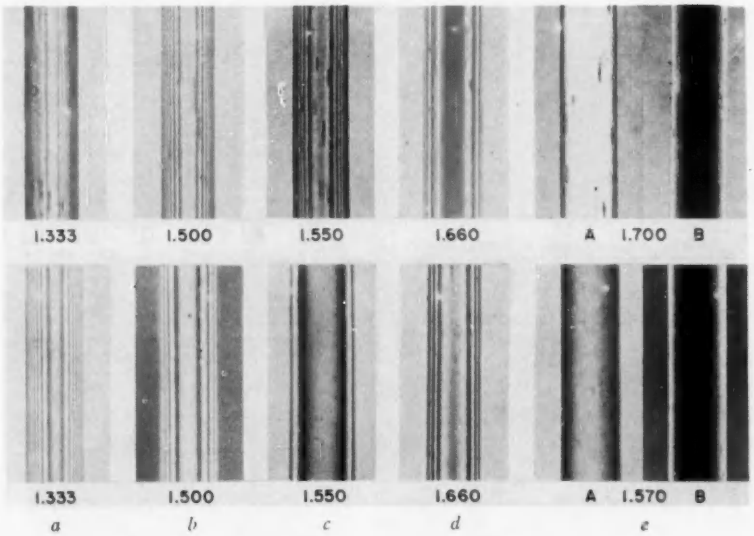


Fig. 3. A Dacron and a nylon fiber under the interference microscope in liquids of different refractive index. Above Dacron, and below nylon. Diameters of fibers are about the same (nylon, 31 microns and Dacron, 28 microns). Refractive index of liquids in (a) 1.333; (b) 1.500; (c) 1.550; (d) 1.660; and (e) 1.700 and 1.570. (Dacron in 1.700 and nylon in 1.570 at two different settings, A and B, of analyzer.) Vibration of light along length direction of fiber, wave length 548 m. μ. 550 ×.

in liquid of $n = 1.700$ and for nylon in liquid of $n = 1.570$, Figure 3). Above this value, fringes appear again.

The number of fringes in birefringent fibers also varies with the orientation of the fiber with reference to the vibration direction of the light which passes through the object. (The direction of the object beam and the reference beam are along the meridian and the equator of the microscope field, respectively. With the Baker microscope, the fiber should preferably be oriented parallel to the meridian to prevent overlapping with the reference area, as discussed under Methods.)

The refractive index of the fiber can be calculated using Equation 1, from the diameter of the fiber, the number of full wave lengths retardation (number of fringes) plus the wave fraction (phase difference measured with the goniometer analyzer) and the refractive index of the medium. The easiest way is to use a medium of a refractive index close enough to that of the fiber, to cause less than one full wave length path difference (no fringes). The following example illustrates this. A nylon filament, 31 microns thick, mounted in a liquid of $n = 1.570$ and parallel to the vibration direction of the monochromatic light of wave lengths 546 m. microns, showed minimum illumination at its center at a goniometer setting of 90°, whereas the medium liquid showed minimum illumination at 240°. The difference of 150° corresponds to a phase difference of 300°, from which a difference in refractive index between medium and fiber ($n_m - n_s$) is calculated, of 0.0145 and a refractive index of the fiber of 1.584[5]. If the same fiber is mounted in a liquid of $n = 1.582$, a phase difference of 30° is found in the same way, corresponding to a difference in refractive index of 0.0015 and the refractive index of the fiber of 1.583[5]. The minimum illumination setting in this measurement was carried out by visual estimation. The accuracy can be greatly increased by using a half shade eye piece.

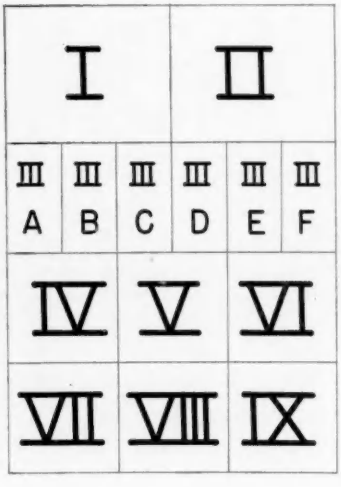
If a cotton fiber is examined, in water, regular fringes are not observed. Fringes are seen only at certain places, especially where the cell wall is in tangential view. In polychromatic light, the fiber shows different and brilliant interference colors. Cell wall and lumen are often clearly differentiated by color contrast, and certain details of the cell wall structure stand clearly out. Especially flat, immature fibers show beautiful color contrast, the cuticle being clearly differentiated from the cellulose wall.

Rayons in longitudinal view show interference colors which vary across the fiber. Due to the noncircular cross-sectional shape, interference fringes in monochromatic light are very irregular in spacing and width.

Observations with Cross Sections

Cross Sections of Viscose Rayons

One of the most important structural details of viscose rayons is the presence of a skin and a core of different density and structure. Many investigators have studied the skin and core structure by



Cross sections of fibers, 6 microns thick, under interference microscope. All figures with shearing objective (except Plate II).

Plate I. Viscose Rayon (Briglo) in water; analyzer, 120 degrees; 550 ×.

Plate II. Viscose Rayon (Briglo) in water; analyzer, 200 degrees; double focus system; 1250 ×.

Plate III. Viscose Rayon (Briglo) in water at different settings of the goniometer analyzer; (a) 0 degrees; (b) 110 degrees; (c) 130 degrees; (d) 150 degrees; (e) 210 degrees; (f) 260 degrees. 550 ×.

Plate IV. Viscose Rayon (Briglo in glycerol; analyzer, 0 degrees; 550 ×.

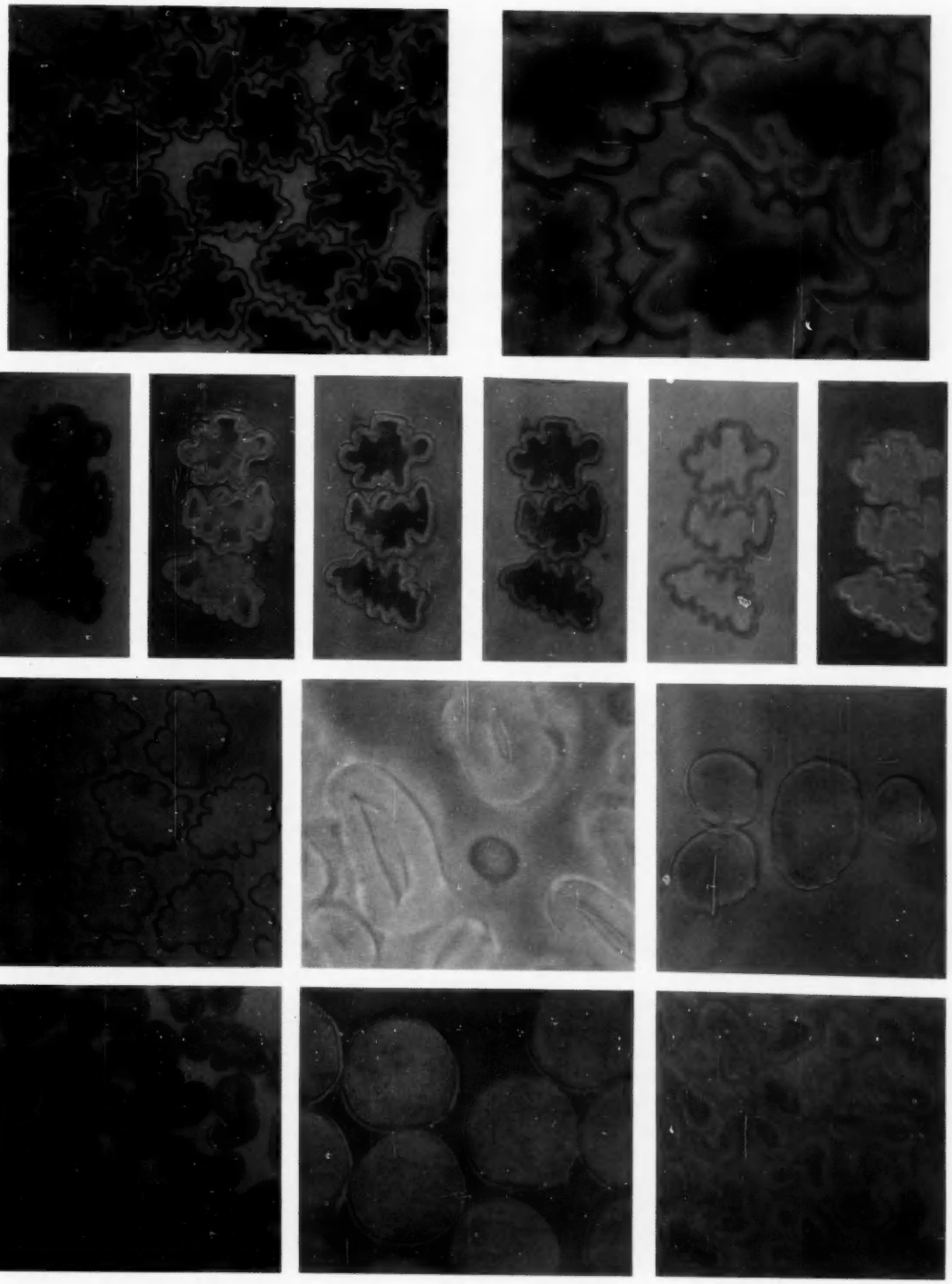
Plate V. Cotton in 11% sodium hydroxide; analyzer, 200 degrees; 835 ×.

Plate VI. Wool in glycerol; analyzer, 50 degrees; 550 ×.

Plate VII. Acetate Rayon in water; analyzer at 90 degrees; 550 ×.

Plate VIII. Nylon in monobromonaphthaline; 550 ×.

Plate IX. Orlon in glycerol; analyzer, 150 degrees; 550 ×.



using differential staining techniques. After Morehead and Sisson [16] had first introduced this method, other investigators have developed modifications (Hermans [7], Rhett Berry [18], and Hara, Sado, and Hashimoto [6]). These methods have been useful in showing not necessarily differences in indexes of refraction so much as showing differences in receptivity to added materials. However, for revealing refractive index differences, the methods have the disadvantage of being rather cumbersome and indirect. The phenomena involved are not fully explained [18]. More direct results have been obtained recently by using the phase microscope (Morehead) [16], Joshi and Preston [11]. Elöd [4] used swelling in combination with the oblique illumination and polarized light. In addition, Joshi and Preston recently obtained interesting results by using defocusing of the microscope. The phenomena which play a part in these cases where staining is not used are rather complicated, however, since reflections of light, scattered in the inside of the fiber by layers of different refractive indices take place as in the formation of the Becke line [9] and as in "central illumination" [10]. Accordingly, regions of higher light intensity (Becke line) are observed at the boundaries of the component layers of different refractive index so that the resolution of the different layers is not fully distinct and sharp. It will be shown in the following how the use of the interference microscope can complement and extend these methods further.

QUALITATIVE OBSERVATIONS. Being careful to observe the experimental conditions, as outlined in Section III, the following additional technique was used. The cross sections were 6 microns thick and cut with the microtome, using paraffin embedding. The paraffin was completely removed and the sections were thereafter mounted in water, without any staining or other treatment. They were studied with the 40 × shearing objective and with the 100 × water immersion double focus objective.

When sections of viscose rayon are studied under these conditions, a skin and core structure is revealed in brilliant color contrast without any further technique, as shown in the color photomicrographs of Plates II and III. The skin and core are clearly distinguished because of differences in the colors. The hue of the colors can be changed almost at will by the setting of the analyzer, and in this way it is possible to obtain perfect contrast in almost any color desired. This is shown in Plate III for six different settings of the analyzer, namely, at 0, 110, 130, 150, 210, 260 degrees using the shearing objective. It is seen from these pictures that the interference colors of skin and core pass through almost all ranges of the Newton scale of interference colors (see Table I).

The main layer of the skin shows small color differences at the regions where the skin folds where two lobes make contact. Preston has already observed differences in birefringence at these places, using the polarized light method, and he explains this as a result of differences in orientation resulting from local compression of the core during the process of coagulation. The orientation would be predominantly tangental at the convex side of the lobe, and radial at the concave side. The variations in color hue observed here corroborate this. A very thin outer layer, which will be termed here "cuticle," of different color will be noted on the outside of the main layer of the skin. This cuticle has also been observed by others [11].

With the double focus 100 × objective, the same section shows similar features. The color contrast is slightly less pronounced. Plate II shows the result at 200 degrees setting of the analyzer. The lower color contrast may be explained by the fact that the reference beam is probably also passing through part of the fiber section. The author obtained the clearest results, when using only a single fiber section for observation with the double focus objective, so as to limit the size of the object.

QUANTITATIVE DETERMINATIONS. The interference colors, described above, permit determination of the path difference by visual estimation by comparing with a color chart or by using a compensator and determining the difference for compensation settings between background and detail.

A few measurements were made using the wedge compensator. When the cross sections are observed under the wedge (placed above the microscope and under an analyzer), the colors range through the same color scale as with different settings of the goniometer analyzer, see Plate III. Small variations in color scale across the microscope field are noticeable even without moving the wedge.

The average of the compensation points of five measurements with 6-microns thick cross sections mounted in water was 1020 m. microns for the core and 1362 m. microns for the skin. The corresponding

TABLE I. Newton's Color Scale

(Modified from Quincke)

No.	Retardation $\lambda = 589$ mμ	Order	Interference colors between crossed Nicols
1	0	0	Black
2	40		Iron-grey
3	97		Lavender-grey
4	158	¼	Greyish blue
5	218		Clearer grey
6	234		Greenish white
7	259		Almost pure white
8	267		Yellowish white
9	275		Pale straw-yellow
10	281		Straw-yellow
11	306	½	Light yellow
12	332		Bright yellow
13	430		Brownish yellow
14	505	¾	Reddish orange
15	536		Red
16	551		Deep red
17	565		Purple
18	575		Violet
19	589	1	Indigo
20	664		Sky blue
21	728		Greenish blue
22	747		Green
23	826		Lighter green
24	843		Yellowish green
25	866		Greenish yellow
26	910	1½	Pure yellow
27	948		Orange
28	998		Bright orange-red
29	1101		Dark violet-red
30	1128		Light bluish violet
31	1151	2	Indigo
32	1258		Greenish blue
33	1334		Sea-green
34	1376		Brilliant green
35	1426	2½	Greenish yellow
36	1495		Flesh color
37	1534		Carmine
38	1621		Dull purple
39	1652		Violet-grey
40	1682		Greyish blue
41	1711		Dull sea-green
42	1744	3	Bluish green
43	1811		Light green
44	1927		Light greenish grey
45	2007		Whitish grey
46	2048		Flesh-red

refractive indices are readily calculated by the formula,

$$n = p/t + 1.333 \qquad (2)$$

in which p is the path difference and 1.333 the refractive index for the mountant (water). The refractive index for core and skin were found to be about 1.50 and 1.55. The determinations are only as accurate as the wedge estimation allows. This rough estimate of the path difference, however, is important in that it gives an idea about the order of the path difference for general orientation preceding more accurate measurements with the goniometer.

More accurate determinations were made with the goniometer analyzer following the technique described under Methods and illustrated in Figure 4. The average of 4 measurements showed a difference in minimum illumination setting between core and medium of 25 degrees, and between skin and medium of 100 degrees. The actual phase differences are therefore 50 and 160 degrees. Since the retardation lies between 2 and 3 full wave lengths, according to the above determinations with the wedge, the actual retardations must be 770 and 880 degrees (2 full wave lengths added). When these retardations are substituted in Equation 1, the refractive index of the core is calculated to be 1.529 and of the skin 1.552.

When the water-swollen sections are mounted in paraffin oil ($n = 1.471$), the retardations are less than one wave length and the refractive indices are found to be 1.512 and 1.537, respectively. When fully dry sections are investigated in paraffin oil, the core and skin are not clearly differentiated and a refractive index of 1.516 is found. This can be explained by the higher micellar orientation in the less swollen conditions.

A comparative study of different types of rayons with the above technique is in progress.

The accurate measurement of the thickness is not too easily achieved. A microscopic check on sections lying sideways, however, gave results close to the 6 microns, at which they were cut, used here for the calculation.

It is possible to short circuit this measurement by mounting the section in fluids of different refractive

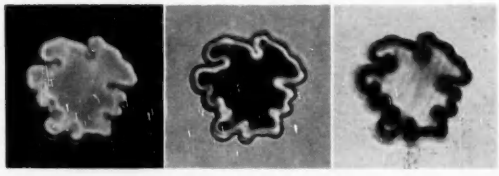


Fig. 4. Measurement of phase differences. Cross sections of viscose rayons, 6 microns thick, unstained under interference microscope, 40 × shearing objective at different settings of goniometer analyzer for minimum illuminator of (a) background at 55 degrees; (b) core at 80 degrees; (c) skin at 135 degrees. Monochromatic light, about 548 m. micron wave length. 560 ×.

indices (n_m and n_m^1). In this case, two equations (1) are obtained

$$\varphi = \frac{360}{\lambda}[(nt)_m - (nt)_s];$$

$$\varphi' = \frac{360}{\lambda}[(n't)_m - (nt)_s]$$

with the refractive index of the detail n_s and the thickness t being unknown so that these two quantities can be calculated. Water and glycerine were found to be suitable mountants for this purpose.

Cross Sections of Cotton and Other Fibers

A brief description of the results obtained with other fibers follows.

COTTON. In cross sections of cotton a beautiful separation of primary and secondary wall is obtained, when they are studied under the proper conditions with this microscope. Plate V illustrates this and speaks for itself.

With monochromatic light, the various components of the fiber can be clearly brought out by setting the goniometer analyzer at minimum illumination for these components. In Figure 5, this is done with a cross section mounted in 18% sodium hydroxide. Minimum illumination of the primary wall occurs at 45°, of the secondary wall at 155° (minimum illumination of the mountant at 190°). Thus, the difference in refractive index of the three components can be qualitatively shown; quantitative studies are in progress.

ACETATE RAYON. The cross sections of acetate rayon show a distinct cuticle on the outside of the fiber, of different color from the rest of the section. The interference color of the central part goes gradually up from the center toward the outside indicating a steady increase of refractive index, as shown in Plate VII. The average refractive index of the main part was determined and found to be 1.44.

NYLON. As a representative of synthetic fibers, cross sections of nylon were examined. If 6-micron thick sections are mounted in water, higher order interference colors result, as can be seen in slightly oblique cross sections, in which the subsequent interference fringes can be seen. Their number is at least four so that we are dealing with at least fifth order interference colors. The order of the colors can be lowered by mounting the sections in, e.g., monobromonaphthalene ($n = 1.657$). Plate VIII shows that in this mountant a good contrast is obtained. A cuticle and also the pits or voids scattered all over the cross section of the fiber are seen in clear color contrast.

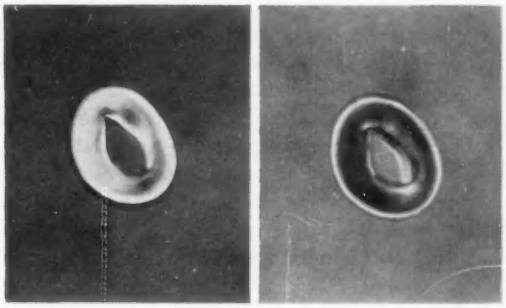


Fig. 5. Cross section of cotton in monochromatic light ($\lambda = 548$ m. microns) under interference microscope, in 18% NaOH. (a) Minimum illumination setting for primary wall at 45°. (b) Same, for secondary wall at 155°. Note also the presence of thin outer cuticle of different refractive index as indicated by different brightness. Magnification 550×.

ORLON. Plate IX illustrates the excellent interference contrast obtained with sections of this fiber. The outer skin portion stands out clearly against the core. The fibrillation and voids are clearly distinguished by color contrast.

WOOL. Plate VI illustrates cross section of wool. Different color hues at the two sides of each section may indicate differences in refractive index.

Conclusion

The author believes that the use of the interference microscope will prove to be of great importance in fiber research for the detailed study of the structure of natural and man-made fibers. For study of refractive index it is superior to other methods in that it allows an immediate qualitative disclosure of even minute differences in refractive index by color contrast and, in addition, an accurate quantitative determination of these indices, regardless of size and location of these details in the fiber.

The principal difficulty with this technique, as pointed out above, is one of careful choice of conditions which may vary from one sample to another. When these conditions are observed, new and useful information is readily obtained.

This technique is a definite improvement over the customary Becke-line method, so far used mostly with fibers [9], in that the latter technique allows only determination of the refractive index of the outermost layer of the fiber which is in direct contact with the medium. The method of central illumination [10] gives only an over-all value and is much less accurate.

Since the refractive index is one of the main optical indicators of the fine structure, the importance of the application of this new method in the study of fiber structure will be clear.

Acknowledgment

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The Yellowing of Nylon

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Abstract

Treatment with thermosetting formaldehyde resins increases the tendency of nylon to yellow when heated. The terminal amine groups of nylon are involved in the discoloration. These amine groups can be blocked by reaction with urea under normal textile curing conditions in order to produce a fabric with less tendency to yellow.

WHITE nylon tends to become yellow during curing and heat-setting operations as well as during consumer use. This appears to be true no matter whether the fabric has been finished to achieve special effects such as stiffness or has been allowed to pass with a minimum of finishing agents.

Perhaps one of the reasons that this problem has not received more attention is the difficulty in measuring the discoloration. Early in the present work instrumental methods of determining discoloration were explored and rejected because the variations in readings generally were greater than the small differences between samples. Consequently experiments were designed so that visual comparison and ranking of samples would provide the desired information.

The Effect of Finishes

In the first experiment, samples of the scoured, white Type 200 nylon taffeta (35% average wet pickup), used in most of the present work, were padded two dips and two nips through solutions containing 8% solids of (1) a low molecular weight urea-formaldehyde resin and (2) a methoxymethylated melamine. Each finish was catalyzed by the presence in the bath of 0.4% diammonium phosphate. Both of these formulations have been used commercially for durable stiffening effects on nylon. A third group of samples was padded through water. As each sample emerged from the pad, it was placed immediately in an oven where it was dried and cured. Conditions of heating for each finish included 2, 5, 10, 20, and 30 min. at 116°, 149°, and 163° C.

After curing, the fabrics were cut to a uniform size, folded into four thicknesses and mounted on a white background. When the samples were judged visually, several conclusions were apparent immediately. First, with each finish the degree of yellowing increases with the time and with the temperature of the cure. Second, the two resins yellow to about the same extent. The differences between samples of the two resins cured at the same time and temperature is less than the difference between adjacent times for either resin at any temperature. Third, the nylon padded through water becomes yellow but not to the same extent as the treated pieces of nylon.

Portions of the fabrics were taken from the pad, air-dried, and stored at room temperature. Two weeks later these samples were cured at 300° F. for 10, 20, and 30 min. These fabrics were about as yellow as the original set cured under the same conditions. This observation is cited to indicate that the laboratory practice of air-drying before curing does not change appreciably the yellowing of the fabrics in this time and temperature range. Air-drying in the laboratory is preferred with the hydrophobic fibers since it guarantees that all fabrics arrive at the curing oven with the same moisture content. The presence of variable amounts of moisture could be a critical factor in some work.

Site of Yellowing

Nylon treated with resin may become a deeper yellow than untreated nylon because the resin itself becomes discolored or because the rate of yellowing of nylon is increased in the presence of the finish. To determine to what extent the resin alone is responsible for the discoloration, glass fabric was "desized" by treatment in warm concentrated sulfuric acid. After thorough rinsing and drying, this fabric was

padded at 10% wet pickup through a catalyzed 28% solids solution of the same urea-formaldehyde resin used before. Thus the applied resin solids was of the same magnitude as on the nylon fabric.

The treated glass fabric was air-dried and portions were cured at 149° C. for 0.5, 2, and 5 hr. In addition part of the fabric cured for 0.5 hr. was put under a strip ironer at 185° C. for 10 min.

On examination it was noted that the resin-treated fabrics did not have the same luster as untreated glass. The only sample, however, which showed any tinge of yellowness was the one which was given the extreme 5-hr. cure. Consequently, it would appear that the distinct yellowing obtained with the resin-treated nylon under less drastic curing conditions could not be due to the finish alone. Moreover, since the yellowing of resin-treated nylon is more pronounced than that of untreated nylon, the yellowing must be the result of an interaction between the nylon and the resin.

Pieces of nylon were padded through (1) a catalyzed solution of 8% solids of the urea-formaldehyde resin and (2) water. Both samples were air-dried and cured at 300° F. for 6½ hr. to exaggerate the discoloration. The fabric treated with resin was very much more yellow than the water control. Using Nickerson's method for hydrolyzing thermosetting resins on cotton [3], portions of each fabric were immersed for 20 min. in a solution of 5% urea and 1.5% phosphoric acid which was maintained at 180-185° F. This procedure appeared successful in removing the thermosetting resin since the treated fabric lost all of its stiffness. There was negligible loss of yellow due to the acid strip, so that the color of the treated fabric remained very much greater than that of the untreated fabric. Since the discoloration is not in the resin but increases in the presence of resin, the yellowing must occur in the fiber at an increased rate due to the interaction with the finish.

Role of Amine Groups

Nylon has three possible reaction sites: the terminal carboxyl groups, the even fewer terminal amine groups, and the multiplicity of amide links. Of the three groups, the amines are most likely to give colored products and therefore their importance to the reaction which produces yellowing of the fabric was investigated first.

The following deamination procedure for nylon was employed. Samples of fabric were placed into 200 ml. of acetic acid and allowed to soak at room temperature for 16 hr. in order to swell the fibers. The acid then was diluted with water to make a 25% solution. Into this stirred bath 200 ml. of 12% solution of sodium nitrite was added over a period of 5 hr. The fabric then was removed from the flask and rinsed thoroughly in running water. As a control, another nylon fabric was treated in the same process without sodium nitrite; this will be referred to as "acid treated."

Portions of the deaminated nylon, the acid-treated sample and untreated nylon were dyed in a bath containing 1% on the weight of the goods of National Alizarine Sapphire BN Conc. 150% (C. I. 1054). The pH of the bath was adjusted to 4.0 with sulfuric acid. According to Peters [4], only the primary amine groups of nylon will be dyed under these conditions.

The dyeings confirmed expectations. The acid-treated sample was darker than the untreated fabric. This was probably due to hydrolysis produced by the acetic acid medium. The sample treated with sodium nitrite solution was very much lighter in color than the untreated indicating that deamination at least to some extent was successful.

Half of each of the undyed portions of the three fabrics was tested for yellowing by treatment with (1) a solution of 8% solids of the urea-formaldehyde resin with 0.4% ammonium chloride and (2) water. The fabrics were air-dried and cured for 1 hr. at 300° F. to accentuate any discolorations. According to the amounts of color, the samples ranked as shown in Table I.

TABLE I

	Sample	Treatment
Least yellow	Untreated	Water
	Deaminated	Water
	Deaminated	Resin
	Acid treated	Water
	Untreated	Resin
Most yellow	Acid treated	Resin

The acid-treated samples are definitely the most yellow indicating that the hydrolysis which provides more end groups also provides more groups capable of yellowing. Deamination definitely decreases yellowing on resin-treated samples. On samples containing no finishing agent, deamination provides a large measure of improvement in color over the hy-

drolyzed (acid-treated) control which should have approximately the same molecular weight as the deaminated sample. This deaminated sample is only a shade darker than the higher molecular weight untreated sample.

Ultraviolet light has a profound effect on polyamides. Achhammer, Reinhart, and Kline [1] reported that the nylon polymer molecules break at the C–N bond of the peptide group creating smaller polymer molecules with the same unit of chemical structure. We have found that following exposure to ultraviolet light there was a decrease in the depth of acid dyeings and an increase in the depth of basic dyeings suggesting a decrease in the number of amine groups and an increase in the number of carboxyls.

The remaining portions of the fabrics from the deamination experiment were exposed in a Fade-O-Meter for 65 hr. The fabrics then were treated and tested as above. The samples ranked in the order shown in Table II.

TABLE II

	Sample	Treatment
Least yellow	Untreated	Water
	Deaminated	Water
	Deaminated	Resin
	Untreated	Resin
	Acid treated	Water
Most yellow	Acid treated	Resin

The ranking of the samples exposed to ultraviolet light is practically the same as that of the previous group with regard to the effect of pretreatment and aftertreatment. As a group, however, the exposed samples were very much more yellow than their unexposed counterparts even though the samples after exposure and before heating are whiter than the original nylon.

The cause of increased yellowing which takes place on heating samples which have been exposed to ultraviolet light was not determined. However, in view of the fact that U.V. exposure appears to reduce the number of free amine groups, one could speculate that the phenomenon might be due to the decomposition of an intermediate which the amines have formed during exposure.

Only the amine groups of nylon will react with acetic anhydride. Therefore to confirm the role of terminal amine groups in the yellowing of nylon, pieces of fabric were soaked in acetic anhydride for 1 min., ½ hr., and 19 hr. The samples then were rinsed free of reactant and air-dried. The dye test showed the sample soaked for the longest time to contain substantially fewer terminal amine groups than untreated nylon. The shorter reaction times had no appreciable effect in reducing the number of amine groups, presumably because of a slow rate of penetration into the fibers. When these fabrics were tested for discoloration, the only sample which showed significantly less yellowing than untreated nylon was the fabric with the reduced amine group content.

Reaction with Urea

Having demonstrated the importance of primary amine groups in the yellowing process, a means of preventing the discoloration was sought. While acetic anhydride is quite effective, its handling properties do not make it desirable for general mill use. Consequently other treatments to retard yellowing were explored.

In theory it should be possible to react the terminal amine groups of nylon with urea. The first attempt to accomplish this was made by maintaining nylon in molten urea for 6 hr. This fabric had a reduced capacity for acid dyes. The fusion then was attempted under less drastic conditions. Nylon was padded through a water solution containing 30% urea. A control piece of nylon was padded through water. Both samples were air-dried and halved. One set of fabrics was cured for 10 min. at 300° F. and the other was cured for 1 hr. at the same temperature. The treated fabrics were rinsed in warm water to remove unreacted urea.

When dyed, the water control cured for 1 hr. picked up slightly less dye than the sample cured for 10 min. The nylon treated with urea and cured for 10 min. was very much lighter in color than either of the water controls. Of the samples treated with urea, the sample cured for 1 hr. picked up slightly less dye than the fabric given the shorter cure.

To determine the optimum concentration of urea, nylon was padded through solutions containing 0, 2, 5, 10, 20, 30, and 40% urea. After air-drying the samples were cured for 10 min. at 300° F. and then rinsed free of unreacted urea. Half of each sample was dyed in a solution of the National Alizarine Sapphire BN Conc. 150% at pH 4.0. The dye pickup decreased as the concentration of the urea in the bath increased. The most distinct changes were produced by 20, 30, and 40% urea. The other half of each

sample was treated with the catalyzed urea-formaldehyde resin and cured for 1 hr. at 300° F. to emphasize color. Whiteness increased with the concentration of the urea treatments starting with 5%.

In the succeeding experiment treatments using 30, 40, and 50% urea solutions were made. The curing times and temperatures are indicated below. Since differences in the color of the dyed samples are more easily detected than differences among the yellowed samples, these fabrics were rinsed in water and then tested only by the dyeing technique. Table III indicates the visual appraisal of the dye pick-up of each sample.

The magnitude of the effect is controlled by the usual variables of concentration, time and temperature. Conditions of curing appear to be more critical than concentration is in the range investigated. The cures are of the same magnitude as are required by many thermosetting resins.

Fabric Properties

When the fabrics treated in solutions of urea became dry, they were very stiff. After the cure and rinse, however, the hand of the fabric was the same as untreated nylon. No major strength changes were evident.

The dyeing characteristics of "ureido-nylon" were explored. There was a slight increase in the depth of dyeing with basic dyes compared with the depth of color on untreated nylon. Dyeings with dispersed colors were unaffected though the gas-fade resistance of these dyes was decreased. The decrease of dye pickup with acid colors was sufficient to suggest the use of "ureido-nylon" in resist effects.

Brief evaluations of the effect and durability of nylon finishing agents, such as polyacrylic and melamine-formaldehyde and urea-formaldehyde stiffening agents, indicated no real difference between applications to untreated and urea-treated nylon, provided, in the case of thermosetting resins containing formaldehyde, that all unreacted urea was first removed by scouring.

ging of Nylon

The effect of the usion with urea on the discoloration of nylon which is not resin finished was studied by measuring the rate of yellowing of untreated and treated nylon. This was done by establishing a discoloration standard.

Samples of nylon then were heated at a given temperature for various lengths of time until by visual comparisons it was possible to estimate the time of

TABLE III

(10 = dark blue; 0 = white)

Temperature of cure	240° F.			300° F.			325° F.				
Time (min.) of cure	25	60	100	5	10	15	1	2	4	6	8
Water	10	10	10	10	9	9	10	10	9	9	10
30% Urea	10	9	8	6	5	5	7	4	3	2	4
40% Urea	10	9	8	4	3	2	6	3	2	3	3
50% Urea	10	9	7	4	2	2	5	3	2	2	2

TABLE IV. Time of Yellowing, Minutes

	Untreated					Treated				
Observer	A	B	C	D	Avg.	A	B	C	D	Avg.
Temperature, °C.										
200	3	3	3	3	3	2	3	2.5	3	2.5
185	6	8	8	7	7	8	10	8	8	8.5
175	10	15	20	13	14	20	20	30	20	22
160	45	60	60	60	56	90	70	90	80	82
150	70	70	70	90	75	100	80	180	140	125
140	240	360	360	300	315	360	360	600	600	480
130	360	480	360	600	450	960	960	1440	1200	1140
120	1680	3360	2100	2100	2310	3360	4800	3360	3600	3780

heating necessary to match the discoloration of the standard. This time was taken as the measure of the rate of yellowing.

From the earlier data on the relative effects of urea concentration and cure, conditions for a mild but effective anti-yellowing treatment could be chosen. Accordingly, the nylon to be treated was padded through a solution of 25% urea, air-dried, cured for 10 min. at 300° F., and rinsed free of unreacted urea. This is a typical treatment; greater differences between untreated and treated fabrics could be produced by altering the conditions of the treatment.

Table IV shows the times to match the discoloration standard prepared by heating a piece of scoured nylon for 3 min. at 200° C. The data are presented graphically in Figure 1.

With the typical urea treatment employed in this experiment, yellowing times are extended by 50%. To extrapolate to lower temperatures, the data were put into the form of an Arrhenius plot (Figure 2) where the times are plotted as logarithms against the corresponding reciprocal of absolute temperature.

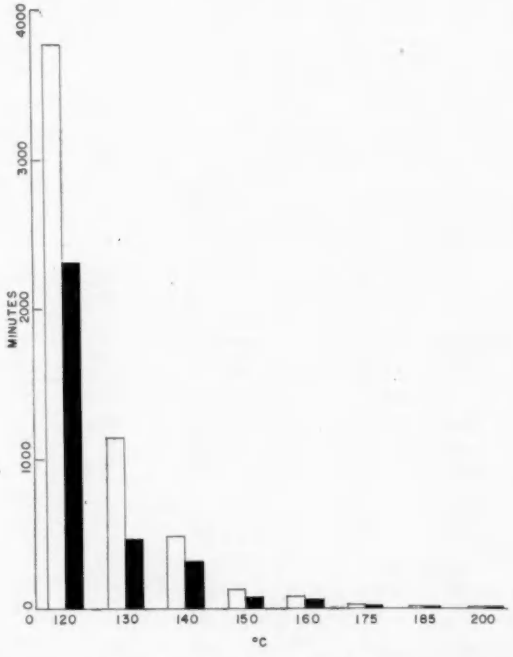

Fig. 1. Comparison of the time of yellowing of untreated (shaded area) and treated (clear area) nylon.

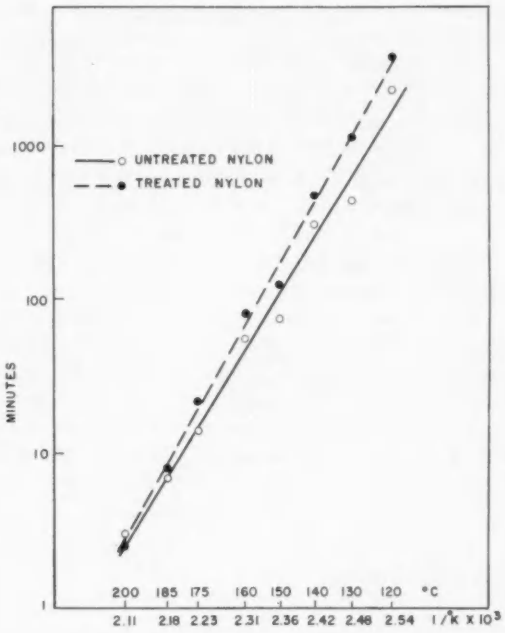

Fig. 2. Arrhenius plot of time of yellowing versus temperature.

Equations for these lines, calculated by a least squares analysis, are

Nylon:

$$\log t \text{ (min.)} = \frac{6530}{T \text{ (° K.)}} - 13.4$$

Ureido-nylon:

$$\log t \text{ (min.)} = \frac{7260}{T \text{ (° K.)}} - 14.9$$

Thus at 100° C., it would take untreated nylon 9 days and "ureido-nylon" 28 days to reach the same degree of yellowing. If this curve could be extrapolated to room temperature (30° C.), the times to reach this same color would be 300 and 2400 years, respectively.

When the fabrics were maintained in boiling water for prolonged periods of time, the time to match the same yellowing standard as before was 7.5 days for nylon and 12 days for "ureido-nylon." The failure of the equations to predict with better accuracy the time in water may be due to the operation of a different mechanism when the heating is carried out in air.

It will be recalled that prolonged heating of scoured nylon caused a slight decrease in the amine group

content as indicated by dyeing experiments. Meacock [2], however, reported a rise of the amine end groups of nylon held at 293° C. in the presence of steam for varying lengths of time. To check on the apparent inconsistency, samples of nylon were heated for 125 hr. at 100° C. in a vacuum and at 106° C. in air. Both samples showed decreased dye pickup indicating that the presence of air is not responsible for the difference in results. A piece of nylon held in boiling water for 260 hr. did show increased affinity for the acid dye.

Thus in the presence of water at high temperatures appreciable hydrolysis apparently occurs. In the absence of water, a different degradative reaction seems to predominate. The existence of two mechanisms is supported by the color of the samples heated in different ways. Samples heated in air were greenish-yellow while the samples removed from the boiling water tended more toward a reddish-brown.

Different Types of Nylon

Through the courtesy of E. I. du Pont de Nemours Co., Inc., samples were obtained of three taffeta fabrics made from (1) Type 100 bright nylon, (2) Type 200 semi-dull nylon, and (3) Type 680 dull nylon. The latter two were received in the greige and portions were scoured in Triton X-100 to remove the finishing oil. Scoured samples of each fabric were padded through a bath of 40% urea, air-dried, cured for 4 min. at 325° F. and rinsed free of excess urea. The time of yellowing was measured by comparing the discoloration after various times of heating with the discoloration of an arbitrary sample. In this case the standard was scoured Type 200 nylon taffeta heated for 6 min. at 200° C.

From the foregoing it is apparent that in dark heat dull nylon takes longer to reach the same degree of

TABLE V. Time of Yellowing

		Observer	A	B	C	D	E	F	Avg.
Temperature	Nylon type	Treatment							
175° C.	100	Scoured	12	16	7	10	8	8	10
"	"	Urea	24	16	20	16	20	10	18
"	200	Greige	8	8	8	7	6	6	7
"	"	Scoured	12	8	10	10	8	6	9
"	"	Urea	28	4	24	6	4	8	12
"	680	Greige	24	16	20	16	24	18	20
"	"	Scoured	32	16	32	24	32	20	26
"	"	Urea	128+	32	48	32	48	40	55+
151° C.	100	Scoured	50	50	30	50	37	35	42
"	"	Urea	300	50	18	110	12.5	50	90
"	200	Greige	40	25	35	25	25	25	29
"	"	Scoured	50	25	100	35	37	30	46
"	"	Urea	150	12.5	125	75	50	30	74
"	680	Greige	175	100	150	125	150	125	138
"	"	Scoured	350	400	175	140	150	150	228
"	"	Urea	800+	200	800+	800+	700	800+	684+
140° C.	100	Scoured	75	100	75	80	75	75	80
"	"	Urea	400	200	175	200	400	300	279
"	200	Greige	75	100	60	65	75	65	73
"	"	Scoured	85	50	85	140	75	60	82
"	"	Urea	300	200	60	80	75	75	132
"	680	Greige	400	200	200	200	200	125	221
"	"	Scoured	2600	200	1600	400	400	600	966
"	"	Urea	4200+	3200	4200+	3200	3200	3600	3600+

discoloration than bright or semi-dull do. The presence of finishing oils on the fabric increases the ease of yellowing. On clean fabric, fusion with urea is effective on all three types of yarn in reducing the tendency to yellow.

Besides the additives to the yarn, the composition of the polyamide polymer also affects the rate of discoloration. For example, a comparison of commercial nylon (a 6, 6 polyamide) with Rilsan (a polyamide 11) indicated that the latter was more prone to yellow. Fusion of Rilsan with urea caused a marked improvement in the resistance of the fiber to discoloration by heat. Patent applications on the treatment of polyamides with urea have been filed.

Acknowledgments

The author wishes to thank Mrs. Marie C. Harkins and Mr. Robert G. Petrella for their assistance in performing much of the experimental work, Mr. Albert C. Nuessle for his continued interest in this study, and the Rohm & Haas Co. for permission to publish these results.

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Manuscript received October 26, 1956.

Summer Program at M.I.T.

In connection with the special summer programs at the Massachusetts Institute of Technology, the Textile Division of the M.I.T. Mechanical Engineering Department is sponsoring two one-week sessions.

July 29 through August 2 will be devoted to the subject, "Impact Behavior of Textile Materials."

"The purpose of the Program is twofold: to familiarize those not active in impact research with current knowledge of impact testing and dynamic behavior of fibrous materials, and to provide active research workers with a forum for discussion of recent advances in this special field. The review lectures will be useful to technologists, physical scientists, and engineers who have not had advanced training or who have been out of school for some time. They will, in addition, provide the necessary background for discussions of current progress in impact studies to be presented later in the Program."

August 5 through August 9 will deal with "Textile Manufacturing Principles." The program will present an engineering approach to the analysis and improvement of textile manufacturing as it is concerned with the conversion of fiber into fabric.

Lectures and discussion sessions will feature both programs. Tuition for each is $150. For detailed information and application forms write to Massachusetts Institute of Technology, Office of the Summer Session, Room 7–103, Cambridge 39, Mass.

National Cotton Council of America

1957 Cotton Research Clinic

The following papers were presented at the 1957 Cotton Research Clinic of the National Cotton Council of America, held February 27, 28, and March 1 in Savannah, Georgia. Other papers presented at the Clinic will appear in the July issue. Reprints of the entire proceedings, bound in a cover, will be distributed through the Cotton Council.

The Effect of Roll Run-Out in Spinning on Yarn Quality

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Introduction

The increasing demand of the consumer for high quality goods at a low cost in today's highly competitive textile market has led cotton mill operators into the field of what might be called "precision manufacturing." This procedure involves careful and consistent checking of all parts of cotton processing equipment so as to establish and maintain maximum tolerances permissible for obtaining a product of desired quality. While there are many moving parts on textile machines that should be included in a complete testing program, the drafting element in a spinning frame possibly has the greatest influence on yarn quality. Within the drafting element itself, the accuracy of the drafting rolls may be of major importance.

During the past few years, many questions have been asked by mill operators regarding the influence of eccentric drafting rolls on yarn quality. How much run-out or eccentricity in drafting rolls on a spinning frame can be allowed before yarn quality is appreciably lowered? Are the run-outs of all the drafting rolls of equal importance? Do higher drafts require lower roll tolerances? What should be the maximum allowable run-outs for new rolls being purchased? Is run-out of drafting rolls more critical for certain drafting systems?

In an attempt to answer some of these questions, the Institute of Textile Technology, about five years ago, undertook a project to review the available literature and to perform some experimental work to evaluate the effects of roll run-out in a spinning frame on yarn quality.

Previous Work

Gregory and Tyson [4] state that within the spinning frame, eccentric drafting rolls are one of the main causes for imperfect drafting. They also claim that this imperfect drafting results from a slight change in the position of the roller nip as the rolls revolve, thus varying the rate of take-up or drafting of the roving. De Marsano [1] also describes how this "nip movement" causes irregular or defective drafting. While the theory of "nip movement" is based on top roll eccentricity, Foster [2] in discussing the theory of Gregory and Tyson states that defective drafting with eccentric bottom rolls also arises in the same manner as with eccentric top rolls.

Gregory and Tyson [4] conclude their discussion by developing formulas which, in essence, state that:

[1] Taken in part from a thesis submitted by Jack O. Middleton in partial fulfilment of the requirements for the M.Sc. degree in Textile Technology by the Institute of Textile Technology, Charlottesville, Virginia, June 12, 1956.

Additional variation introduced to a yarn due to roll eccentricity is proportional to $\frac{\text{eccentricity} \times \text{draft}}{\text{roll circumference}}$

The draft referred to here is that in the draft zone immediately behind the eccentric roll and not the total draft where there are several draft zones.

In a later article, Foster and Tyson [3] present results to further substantiate the formulas. Their data indicate that, in a good average yarn, when the additional variation introduced to a yarn by top roll eccentricity reaches 8%, a loss of 5% in yarn skein strength results.

In addition to the nip movement caused by an eccentric bottom roll, there is another factor which must be considered, and that is variation in surface speed. In the case of an eccentric top roll turning on a round bottom roll, the top roll, while being driven by contact with the bottom roll, will have a relatively constant surface speed. In this case nip movement is probably the major contributing factor to defective drafting. Such is not entirely true for an eccentric bottom roll. Since the bottom roll is driven and has a constant angular velocity, the speed of a point on the circumference will vary as the effective radius changes. This variation in surface speed causes a variation in the amount of stock delivered during one revolution of the bottom roll. Nip movement also exists when the bottom roll is eccentric and it is logical to assume that nip movement and varying surface speeds could work together in causing defective drafting. There is thus an indication that bottom front roll eccentricity can be even more detrimental to yarn quality than top roll eccentricity.

Since the investigations reported in the literature are based on studies of top roll eccentricity, it was decided to conduct experiments using the bottom rolls of a spinning frame to determine the effects of roll run-out on yarn quality.

Experimental and Results

The first series of tests were conducted to obtain typical results [5]. The study was made on a Saco-Lowell Roth long draft spinning frame using a draft of approximately 18.75 while spinning 30's warp yarn from single creeled 1.60 hank roving. A picker lap produced from $1\frac{3}{32}$ in. Middling Delta cotton was processed into roving and eight bobbins of roving were selected for their similarity in uniformity.

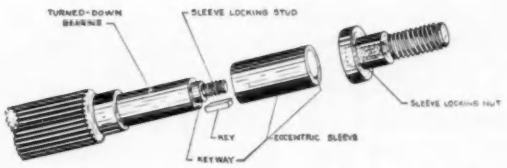

Fig. 1. Schematic drawing showing the manner of attachment of eccentric sleeves to the bottom roll of the spinning frame.

The eight bobbins of roving were spun into yarn on eight spindle positions. Ten doffs of yarn were produced, with the amount of run-out in the front bottom steel roll of the spinning frame being changed for each doff. For the first series of tests, a new front roll was purchased and used as received. For subsequent doffs of yarn, the roll was machined down at the bearing sections to permit the use of eccentric sleeves to introduce various run-outs. The modification of the roll is shown in Figure 1. Measurements of the amount of run-out at each spindle position were made by using a dial indicator gauge mounted on a magnetic base. The range in the deflection of the needle of the gage as the roll turned was recorded as run-out in thousandths of an inch. The method of making the measurements is shown in Figure 2. The determinations were made by turning the spinning frame by hand to avoid excess deflection of the needle due to machine vibration at operating speed.

It was found that the new roll varied in the amount of run-out before the eccentric sleeves were mounted,

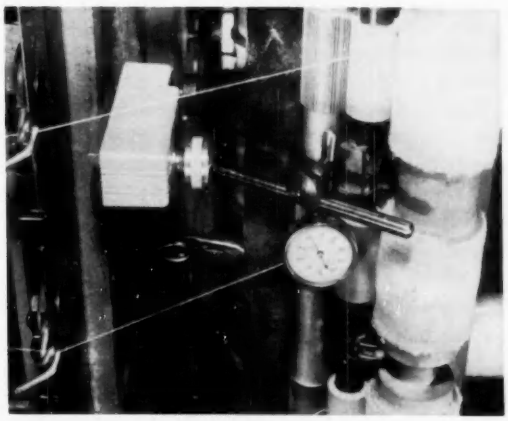

Fig. 2. Photograph showing the method of determining the amount of run-out in the front bottom roll of the spinning frame.

the average value being 0.003 in. Sleeves were machined to produce 0.003, 0.006, 0.009, 0.012, 0.015, 0.018, 0.021, 0.025 and 0.030 in. of run-out on the lathe. However, it was found that the roll produced slightly different amounts of run-out at the fluted sections within each set of eccentric sleeves. This was probably due to the original eccentricity of the fluted sections relative to the bearing sections of the roll itself.

The yarn spun at each doff was tested for per cent nonuniformity, using the Brush Uniformity Analyzer at a unit length of 0.5 in. and a 10-ft. sensitive length for skein and single-end strength. The results obtained are shown in Figures 3, 4, and 5. It will be noticed that good correlations exist between front bottom roll run-out and both per cent nonuniformity and strength of yarn. As greater amounts of run-out were introduced in the roll, yarn quality progressively deteriorated. For the particular cotton, spinning system and draft used in these tests, the point at which per cent nonuniformity increased and strength of yarn began to decrease rapidly appeared to be at about 0.007 in.

Later a second series of experiments was conducted to more fully investigate the effects indicated by the first series of tests [6]. In this study, the tests were designed to determine the effects of bottom steel roll run-out in a spinning frame on yarn quality over a range of drafts and to investigate the influences of run-outs of the front, middle, and back bottom rolls on yarn properties. The experiments were carried out on a Saco-Lowell Duo-Roth spinning frame, using $1\frac{3}{32}$ in. Middling California cotton. A supply of 1.00 hank roving was prepared by processing cotton from laps into roving using identical conditions of carding, drawing, and roving.

Each bottom roll in the spinning frame was fitted with eccentric sleeves in a similar manner to that previously described in Figure 1. The run-out at each fluted section was also measured in the same manner as used in the earlier experiments. The four yarn counts produced included 18's, 25's, 32's, and 40's which were spun from the 1.00 hank roving under the three following conditions:

1. With middle roll run-out held constant at an average value of 0.007 in. and back roll run-out held constant at an average value of 0.0035 in., the bottom front roll run-out was varied by use of eccentric sleeves of 0.003, 0.006, 0.009, 0.014, and 0.025 in. run-out.

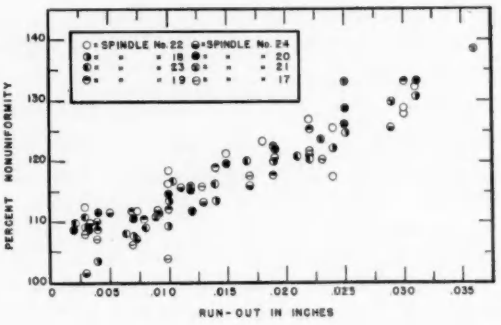

Fig. 3. The relationship between front bottom roll run-out and yarn per cent nonuniformity (30's yarn—Roth spinning).

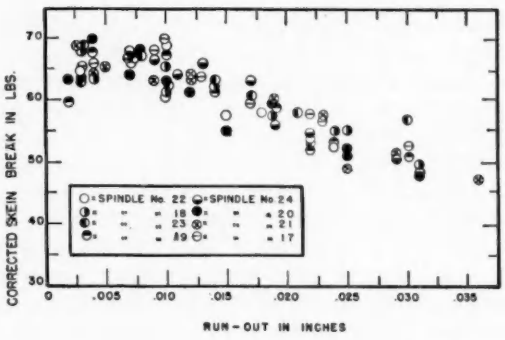

Fig. 4. The relationship between front bottom roll run-out and yarn skein breaking strength (30's yarn—Roth spinning).

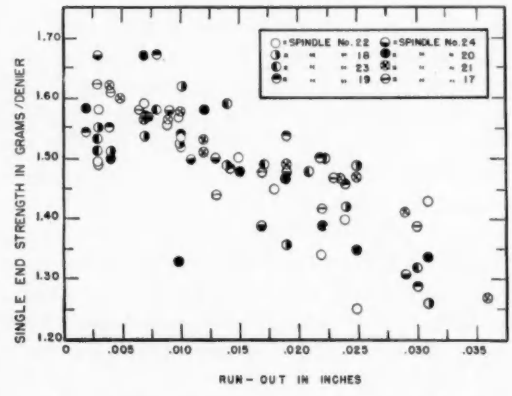

Fig. 5. The relationship between front bottom roll run-out and yarn single-end breaking strength (30's yarn—Roth spinning).

2. With front roll run-out held constant at an average value of 0.0025 in. and back roll run-out held constant at an average of 0.0035 in., the bottom middle roll run-out was varied by use of the same eccentric sleeves as listed for the front roll.

3. With front roll run-out held constant at an average value of 0.0025 in. and middle roll run-out held constant at an average value of 0.007 in., the bottom back roll run-out was varied by use of eccentric sleeves with the same run-outs as listed for the front roll.

The yarn spun at each test condition was tested for per cent nonuniformity, skein, and single-end strength. The per cent nonuniformity measurements were made on a Brush Uniformity Analyzer. In preliminary yarn tests made on the Analyzer at 10-, 1-, and 0.4-ft. sensitive lengths, it was found that the 1-ft. sensitive length best showed the yarn variations being studied and this was accordingly used for all the test yarns reported in this series.

The effects of bottom front roll run-out on the skein strength of 18's yarn are shown in Figure 6. Similar trends were apparent in the other yarn counts studied, that is, a decrease in yarn skein strength as front roll run-out increased. For single-end strength, the effects of the front bottom roll run-out were somewhat more scattered. The results of the tests are illustrated by the 25's yarn, Figure 7. The same general relationship was evident in the other yarn numbers studied. Despite the scatter the trends of reduced strength with increased roll run-out are statistically significant. An example of the correlation between yarn per cent nonuniformity and bottom front roll run-out is shown in Figure 8, where the results are plotted for the 32's yarn. Again, as roll run-out increased, yarn per cent nonuniformity deteriorated for all four yarn counts.

Varying the amount of run-out in the bottom middle roll was found to have little or no effect on yarn quality. A typical example of the results obtained is shown in Figure 9, where roll run-out is plotted against the skein strength of 40's yarn. The

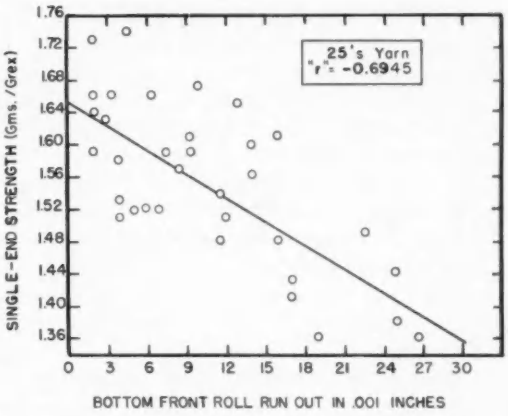

Fig. 7. The relationship between front bottom roll run-out and yarn single-end breaking strength (25's yarn—Duo-Roth spinning).

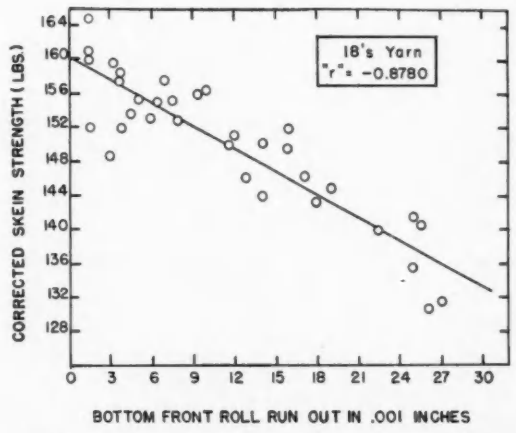

Fig. 6. The relationship between front bottom roll run-out and yarn skein breaking strength (18's yarn—Duo-Roth spinning).

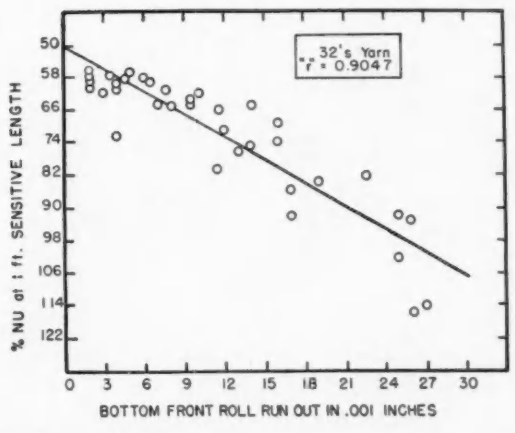

Fig. 8. The relationship between front bottom roll run-out and yarn per cent nonuniformity (32's yarn—Duo-Roth spinning).

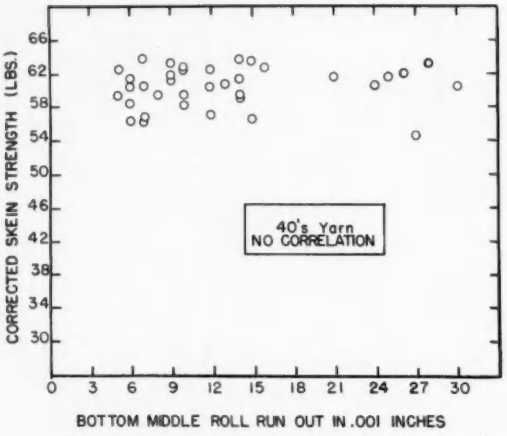

Fig. 9. The relationship between middle bottom roll run-out and yarn skein breaking strength (40's yarn—Duo-Roth spinning).

results may be explained by the fact that the low break draft of 1.38 used in these experiments was not enough to show up the variations introduced by the eccentric middle roll.

Practically no effects on yarn quality were found for the four yarn counts produced when the bottom back roll run-out was increased up to 0.026 in. An example of the results is given in Figure 10, where roll run-out is plotted against the skein strength of the 40's yarn. Here again, the fact that little or no drafting takes place behind the back roll probably accounts for the lack of effect of back roll run-out.

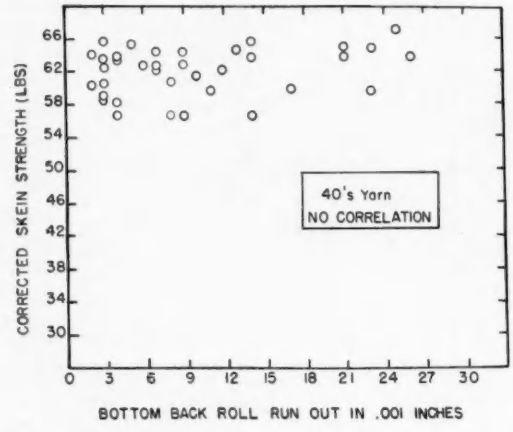

Fig. 10. The relationship between back bottom roll run-out and yarn skein breaking strength (40's yarn—Duo-Roth spinning).

Discussion

From the study of front bottom roll eccentricity, the percentage losses in skein breaking strengths of the four yarn counts were determined, Figure 11. In a similar manner, the percentage increases in per cent nonuniformity were also calculated for the four yarns and are shown in Figure 12. The effect noted for both strength and per cent nonuniformity was that as the front zone draft increased from 13 to 30, deterioration in these yarn qualities increased. These relationships would seem to confirm the conclusions of previous investigators that increases in draft cause increases in the variation introduced in yarn by roll eccentricities. However, in the experiments described, a different yarn number was produced at each draft. Additional work should be performed to determine whether the amount of draft or the weight of the strand of fibers being drafted is the controlling factor.

In considering the results shown in Figure 11, it is interesting to refer to Sheldon's [7] formula for predicting skein strength of carded yarns. According to his calculations, a loss of 11% in break factor results from a decrease of $\frac{1}{16}$ in. in staple length of cotton. In the case of the 40's yarn in Figure 20, the breaking strength of the yarn is decreased approximately 11% when the run-out in the front bottom roll in the spinning frame increases from about 0.003 in. to about 0.010 in. The yarn produced with the front roll run-out of 0.010 in. is no stronger than would be a yarn spun from a $\frac{1}{16}$ in. shorter cotton with 0.003

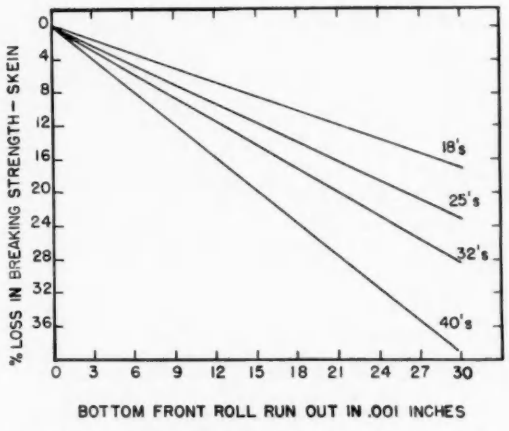

Fig. 11. The relationships between front bottom roll run-out and loss in yarn skein breaking strength for four yarns (Duo-Roth spinning).

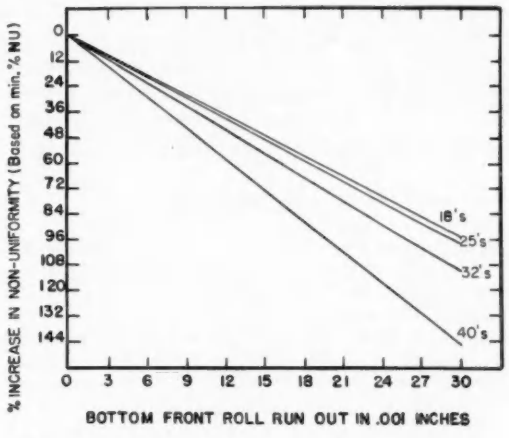

Fig. 12. The relationships between front bottom roll run-out and increase in yarn per cent nonuniformity for four yarns (Duo-Roth spinning).

in. run-out in the front roll. The price of cotton is based, among other things, on staple length. It would seem economically unwise to lose the premium paid for extra staple length in mechanical defects in equipment, such as roll run-out.

Some questions may arise as to why yarn strength and uniformity did not seem to be affected in the first series of tests until the front bottom roll run-out reached 0.007 in., Figure 3, 4, and 5, while in the second series yarn properties appeared to deteriorate with smaller run-outs, Figure 6, 7, and 8. The only plausible explanation for this is that different spinning systems were used in each of these studies. The Roth spinning system was used for the initial experiments, whereas the Duo-Roth drafting arrangement was studied in the second series of tests. It is believed that the Duo-Roth double apron drafting system better controls the fibers entering the nip of the front rolls. Due to this better fiber control, the Duo-Roth system may be more sensitive to small increments of roll run-out than is the Roth drafting element.

As a final illustration of the effects of bottom front roll run-out on yarn quality, two yarn appearance boards are shown in Figure 13. The two yarns were spun from the same bobbin of roving on the same spinning spindle. The yarn on the left was produced when the roll run-out was 0.027 in., the one on the right when the run-out was 0.002 in. While this example illustrates a rather extreme case, it shows the type of yarn variation resulting from any excessive amount of roll run-out.

Conclusions

Within the limitations of the cotton, equipment, yarn numbers and drafts used in these experiments, the following conclusions may be drawn:

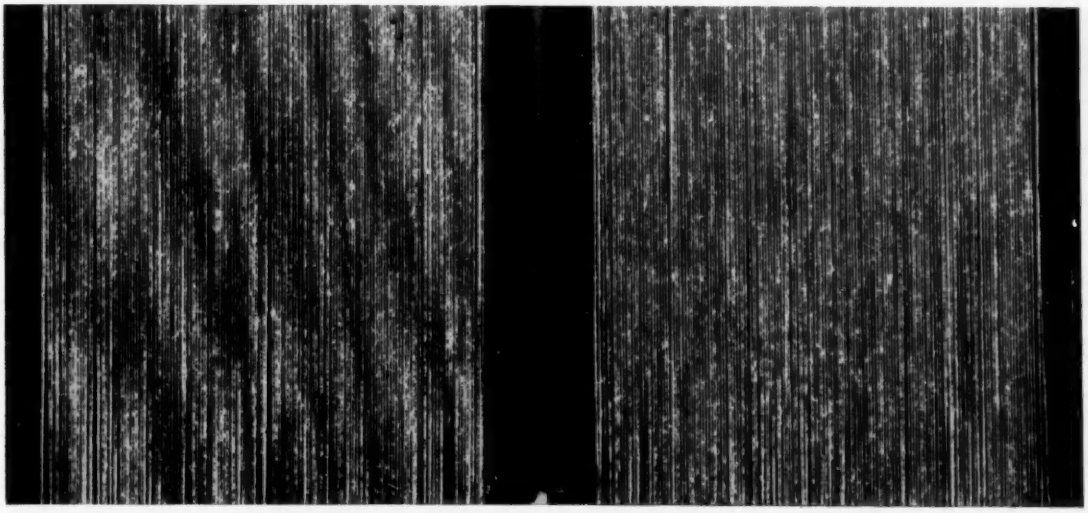

Fig. 13. Photograph showing the effects of excessive front bottom roll run-out on yarn appearance (30's yarn—Roth spinning).

1. Back bottom spinning roll run-outs up to 0.025 in. have no effects on yarn per cent nonuniformity or strength.

2. Middle bottom spinning roll run-outs up to 0.025 in. have no effects on yarn per cent nonuniformity or strength.

3. As run-outs in the front bottom spinning roll increase, yarn per cent nonuniformity increases and strength decreases.

4. As the amount of draft in spinning increases, the effects of front bottom roll run-out on yarn quality become more pronounced. Additional work should be done, however, to separate the influences of draft and weight of stock being processed.

5. There is evidence to indicate that the effects on yarn quality of small amounts of run-out in the front bottom roll may vary for different types of spinning systems.

Summary

The results of the experiments indicate that, while middle and back bottom roll eccentricities are not critical factors affecting yarn quality, the amount of run-out in the front bottom roll can significantly lower yarn strength and uniformity. The deterioration in yarn quality takes place more rapidly as the amount of draft increases. While it is difficult to list specific limits for front roll run-out, it should be possible for a mill to establish its own tolerances taking into account the amount of draft being used and the level of yarn quality desired.

As a final recommendation, it is believed that checking of front roll eccentricity in spinning should prove of value to any mill. As a minimum schedule, it is suggested that the checking should be done whenever a new roll is purchased or installed and also at the time of periodic overhaul of the spinning frame.

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The Effect of Processing on Cotton Fiber Properties

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Abstract

In order to ascertain the effect of several processing steps on cotton fiber mechanical properties, six cottons in one-bale quantities were manufactured under identical conditions into a standard construction sheeting. Samples were removed at several processing stations including the original bale, the yarns, the scoured fabrics, the bleached and mercerized fabrics as well as resin-finished fabrics. The mechanical properties of fibers taken from these stages were determined and the effect of each processing step was examined. It was noted that cotton fiber properties are altered by processing and that the magnitude of the changes in these properties is not constant for all cottons. In many cases the changes in fiber properties are functions of the original fiber properties. It was also shown that the changes in fiber properties are reflected in the changes of the fabric characteristics, thus indicating the validity of the premise that fiber properties influence the behavior of cotton fabrics.

Introduction

Research in the field of cotton has been directed mainly toward the evaluation of the characteristics and quality of the fiber and the relationships of those fiber characteristics to processing efficiency and end-product performance. Testing techniques have been developed which allow the cotton buyer and spinner to evaluate fiber characteristics so that a certain level of quality can at all times be maintained. Cotton fiber research has not been limited to the evaluation of bundle properties by such instruments as the Micronaire, Arealometer, Stelometer, Speedar, and Pressley Strength tester, since recently the evaluation of single fiber mechanical properties has become an accepted technique of fiber research. The methods of single fiber research have been adequately described in previous publications [9, 10, 14]. The direction of current cotton fiber research can be well summarized as follows: To ascertain in what manner cotton properties and characteristics influence processing efficiency and yarn and fabric performance. While not all cotton research can be considered to fall under this general theme, the great majority of effort in cotton research does attempt to provide parts of an answer to this broad and fundamental question.

Recent work at Textile Research Institute has followed this pattern. Single fiber properties have been shown to relate to roving, sliver, and yarn characteristics [12]; the effect of fiber fineness on processing efficiency and end-product performance has been indicated [8]; fiber length, and more important the fiber length distribution, have been shown to effect processing efficiency and end-product performance [13]. Other workers have established relationships between cotton fiber properties and yarn characteristics [3, 4, 6, 7, 15]; while some current research at TRI, sponsored by the U.S. Department of Agriculture, has shown that cotton fiber properties influence final fabric characteristics such as breaking strength and elongation, abrasion resistance, and fabric stiffness. From this short review, therefore, it is clear that a great deal of research effort has been and is still being expended in order to establish the influence of cotton fiber properties on the processibility and end-product quality of cotton.

A somewhat different approach, which in effect attempts to answer the same fundamental question stated above, has recently been undertaken at TRI. Textile processing steps, such as picking, carding, spinning, weaving, mercerizing, and resin-finishing can be expected to affect the cotton fibers. In performing these textile operations, whether they be mechanical or chemical, stresses are exerted on the fibers and the properties of the fibers are correspond-

ingly altered. It is easy to visualize that during carding and spinning, for instance, fibers are extended and some of the weaker fibers may be broken. Fiber kinkiness is partially removed during these parallelizing operations, and various other changes can be predicted to be caused by any given textile operation. Many of these changes in fiber characteristics are desirable, while certain others are merely a necessary evil. Thus the effect of mercerizing and resin-finishing on fiber properties is probably a beneficial and desirable one, whereas in general the mechanical operations exert a change in the fiber properties which is not necessarily beneficial and may even be decidedly harmful. This paper presents the results of a study aimed at determining how cotton fiber properties are altered during the processing operations. The purpose of this research is threefold: (1) to show the manner in which fiber properties are altered by textile processing, both mechanical and chemical, (2) to show that the changes in fiber properties caused by some operations are not constant, but that they are a function of the original cotton fiber properties, and (3) to show that, due to most treatments, changes in fiber properties are reflected in the change of the fabric property. Previous work in this field has been restricted to textile operations preliminary to weaving, and indicates that changes in mechanical fiber properties are caused by carding, roving, and spinning [1, 5, 11, 12]. It has also been reported that cotton cellulose becomes chemically degraded by mechanical processing [16], although other workers have contradicted this point of view [2]. Changes in mechanical properties of cotton due to a formaldehyde cross-linking treatment have been reported [17].

Experimental

As part of a different research project at TRI, six cottons in one-bale quantities were manufactured into a standard construction sheeting at the Pilot Plant of Dan River Mills. The processing outline that was followed was considered to be optimum for each cotton and was carried out under identical conditions for all six cottons. A brief outline of the processing operations is presented in Table I. The six cottons were chosen for their unique combination of fiber properties which are listed in Tables II and III. During the manufacturing process, samples were taken at all processing stations and the fiber properties were carefully evaluated. For the purposes of this study the mechanical properties were evaluated on fibers taken from the bale, the yarns, the scoured fabric, bleached and mercerized fabric, and from fabric that had been bleached, mercerized, as well as resin-finished. Fifty fibers from each cotton were removed from each one of these stages and the mechanical properties were determined by previously described methods. In order to be able to relate the properties of the fibers from the several fabrics to the properties of the corresponding fabric, the properties of these

TABLE I. Processing Outline

1. Opening
2. Picking
3. Carding
4. Drawing (two operations)
5. Roving (1.5 hank, optimum twist for each cotton)
6. Spinning
   - 21/1 at 4.75 TM
   - 29/1 at 4.75 TM
   - 13/1 at 3.45 TM
7. Weaving (sheeting 65×64 construction 21/1 both warp and filling)
8. Singeing
9. Desize (enzymatic)
10. Scour
11. Bleaching (hydrogen peroxide)
12. Mercerizing (52° Twaddell sodium hydroxide)
13. Resin treatment (3.5% cyclic ethylene urea on weight of goods)

TABLE II. Bundle Fiber Properties

	Micronaire, μg/in.	Mean length array method, in.	Arealometer, mm.²/mm.³ "A"	Arealometer, mm.²/mm.³ "D"	Pressley strength 5.0-mm. gauge, g./grex
Pima	3.73	1.26	505	27	2.42
Deltapine	3.55	0.84	536	52	1.34
Sea Island	3.60	1.30	521	47	2.03
Acala	4.45	1.08	439	23	1.71
Hybrid	4.04	1.23	474	34	2.28
Lengupa	5.65	0.88	358	20	1.65

TABLE III. Single Fiber Mechanical Properties in Bale Stage

	Breaking stress, Mg./cm.²	Breaking elongation, %	Elastic modulus, Mg./cm.³	Uncrimping energy per unit vol. ×10⁻³ g./cm.²
Pima	6.55	11.0	66	0.80
Deltapine	3.54	6.6	52	1.22
Sea Island	5.10	8.8	73	0.93
Acala	4.25	7.9	52	1.18
Hybrid	6.01	6.0	100	0.95
Lengupa	5.10	12.4	43	1.20

were also determined. Breaking strength and elongation were used as indices of the fabric characteristics, and the following four fiber properties were considered: single fiber breaking stress, single fiber breaking elongation, single fiber elastic modulus, and single fiber uncrimping energy.

Results

Certain generalizations can be made as to the changes in fiber properties due to the processing operations. Table IV presents the four fiber properties under consideration in the several processing stages as averages of the six cottons. One might conclude from this table that with some notable exceptions the fiber properties are not greatly altered by the processing operations. The average fiber breaking stress is decreased significantly only by resin-finishing. Fiber breaking elongation decreases steadily throughout the entire processing line as would be expected in view of the tensions exerted on the fibers by all textile operations. The average fiber elastic modulus alternately increases and decreases, although within experimental error it might be concluded that the average elastic modulus is not affected by processing steps subsequent to spinning. There is a large increase in fiber stiffness (elastic modulus) from the bale to the yarn stage and this too is to be expected in view of the tensions exerted on the fibers during carding, roving, and spinning. While tensions are also exerted on fibers in the finishing operations, these are carried out in the wet state where the relaxation of the fibers is possible. Fiber uncrimping energy decreases from bale to yarn as expected; again this is due to the tensions exerted on the fibers during processes preliminary to and including spinning. Uncrimping energy increases sharply when fibers from fabrics are examined and this is due to the superimposition of fabric crimp over the natural fiber crimp. The very large increase in fiber crimp due to resin-finishing indicates that a considerable portion of the applied resin is deposited on the fiber surface thus increasing the bending stiffness or modulus of the fibers.

In averaging the fiber properties of the six cottons at the several processing stages a considerable amount of important information is obscured. Considering the changes in fiber properties from the bale to the mercerized and bleached fabric, an increase in the fiber elastic modulus from 64 to 87 Mg./cm.^{2} for the six cottons is observed (Table IV). However, when the six cottons are considered individually it is seen that the different cottons respond to a different degree to the bleaching and mercerizing treatment. Figure 1 shows the change in the fiber elastic modulus for the six cottons as a function of the original fiber elastic modulus. It must be pointed out that these values indicate changes in fiber properties due to all operations preceding and including bleaching and mercerization. It can be seen that the low elastic modulus cottons increase their elastic modulus to a

TABLE IV. Fiber Properties at Several Processing Stations. Averages of Six Cottons

	Fiber breaking stress, Mg./cm.^{2}	Fiber breaking elong., %	Fiber elastic modulus, Mg./cm.^{2}	Fiber uncrimping energy per unit vol. $\times 10^{-3}$ g./cm.^{2}
Bale	5.09	8.8	64	1.04
Yarn	5.30	8.4	82	0.79
Scoured fabric	5.45	7.8	80	1.52
Bleached and mercerized fabric	5.09	6.9	87	1.85
Bleached, mercerized and resin-fin. fabric	3.38	4.6	76	3.95

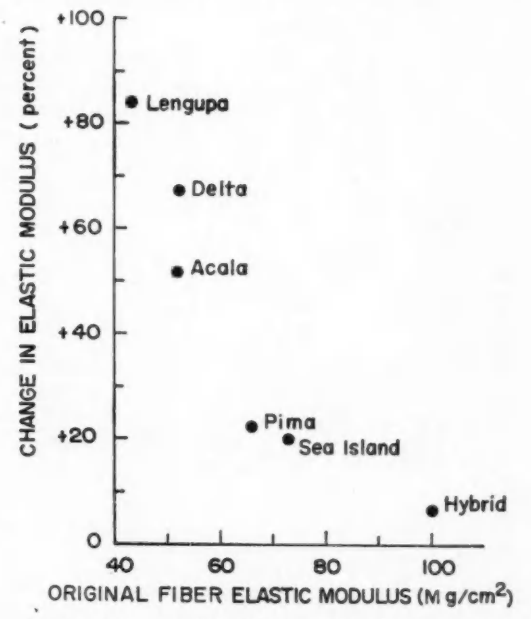

Fig. 1. Change in fiber elastic modulus due to processing (through mercerization) as a function of original fiber modulus.

much greater extent than the cottons with an originally high elastic modulus. Thus, the fractional increase in fiber elastic modulus is an inverse function of the original fiber elastic modulus; and the differences among the six cottons tend to be leveled out.

The breaking stress averaged over the six cottons remains unaltered from 5.09 Mg./cm.^{2} in the bale stage to 5.09 Mg./cm.^{2} in the bleached and mercerized condition (Table IV). This in effect indicates that all the processing steps up to and including mercerization have no effect on fiber properties. However, considering each one of the six cottons separately and plotting the fractional change in fiber breaking stress versus the original fiber breaking stress, it can be seen in Figure 2 that there is a pronounced change in fiber breaking stress and that this change is a function of the original breaking stress. The three weaker cottons increase in breaking stress, whereas the three stronger cottons decrease in breaking stress.

Similarly, while the average breaking elongation for the six cottons (Table IV) shows a decrease from bale to the bleached and mercerized fabric, examination of Figure 3 shows that this decrease is also a function of the original fiber breaking elongation. Cottons with a high breaking elongation exhibit a greater decrease in this property than cottons with a low breaking elongation.

Thus, the cumulative effect of all processing up to and including mercerization is a leveling-out effect. The differences among the six cottons in the bale stage are reduced when fibers from the mercerized fabric are considered.

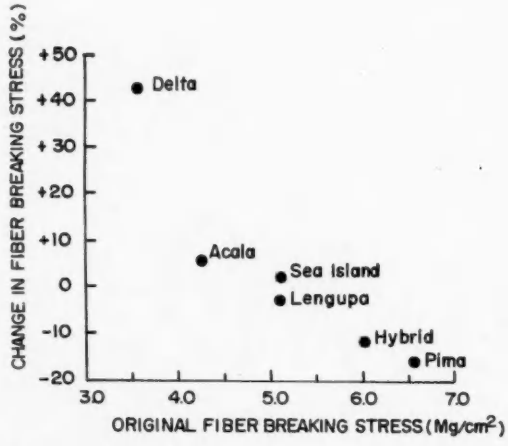

Fig. 2. Change in fiber breaking stress due to processing (through mercerization) as a function of original fiber breaking stress.

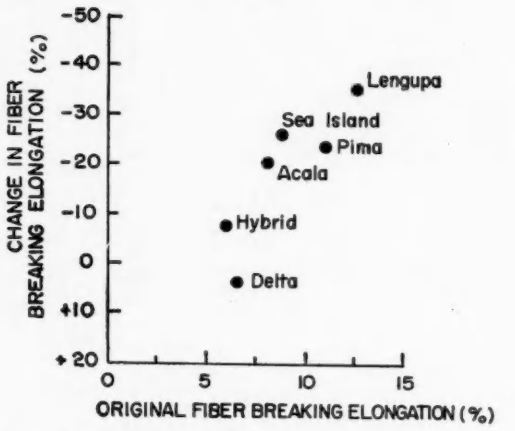

Fig. 3. Change in fiber breaking elongation due to processing (through mercerization) as a function of original fiber breaking elongation.

The final treatment, that of resin-finishing, does not follow the pattern described for mercerization. The effect of resin-finishing on fiber properties is to decrease breaking stress, breaking elongation, and elastic modulus and to increase considerably the fiber uncrimping energy. It appears that the magnitude of these changes is practically constant for the six cottons and is not a function of the fiber property before the resin treatment.

The effects of the processing operations are illustrated in another manner in Figures 4 to 7. The actual fiber breaking stress values at the several processing stations are plotted in Figure 4. The six cottons are divided into three groups of two, that is, a high strength group (Pima and Hybrid), an intermediate strength group (Lengupa and Sea Island) and a low strength group (Acala and Deltapine); and the manner in which the fiber breaking stress is altered by processing is indicated. The fiber breaking stress is decreased for the high strength group, remains essentially unaltered for the intermediate strength group, and is increased for the low strength group by the processing operations up to mercerization. Thus, the large spread in fiber breaking stress values in the bale stage is virtually eliminated in the bleached and mercerized fabrics. The large decrease in fiber breaking stress due to resin-finishing is the same for each of the three groups. It is interesting to note the parallelism between the fiber and fabric

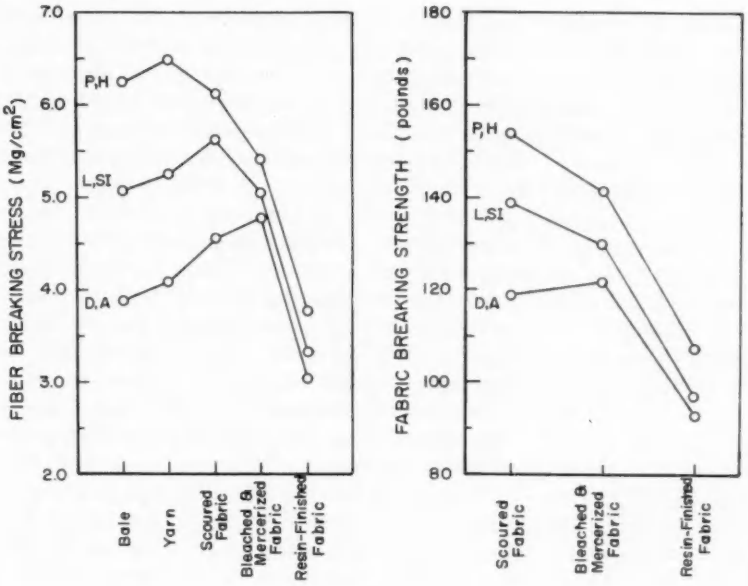

Fig. 4. Effect of processing on fiber and fabric strength properties. P—Pima, H—Hybrid, L—Lengupa, SI—Sea Island, D—Delta, A—Acala.

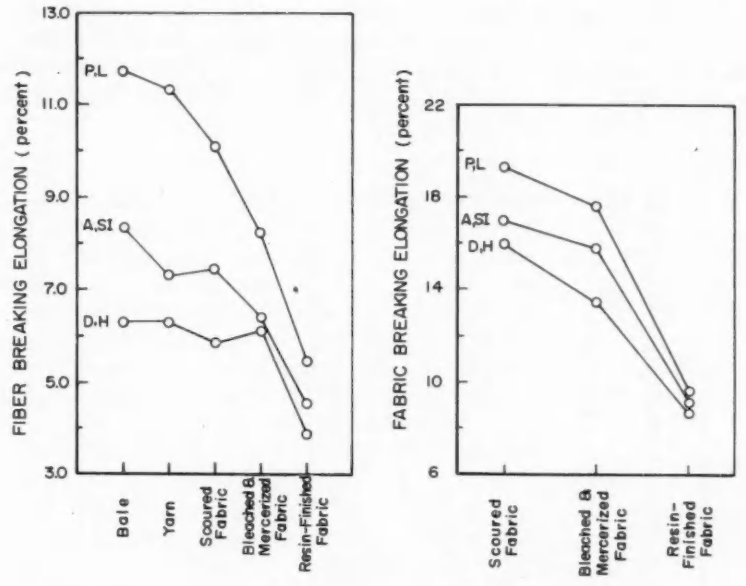

Fig. 5. Effect of processing on fiber and fabric breaking elongation. P—Pima, L—Lengupa, A—Acala, SI—Sea Island, D—Delta, H—Hybrid.

properties. Also in Figure 4 is plotted the fabric breaking strength of the sheeting made from the cottons in each of the three strength groups. The first point to note is that the relative positions of the three groups is the same for the fabric breaking strength as for the fiber breaking stress. The combined effect of bleaching and mercerizing is to decrease the fabric breaking strength of the two stronger cotton groups, but to increase the fabric breaking strength of the low strength cotton group. This behavior was previously observed when the fiber breaking stress values were considered. Resin-finishing decreases fabric

breaking strength to the same extent for the three cotton groups and this too was previously observed in the fiber breaking stress values. This parallelism between fiber and fabric strength properties clearly indicates the influence of fiber properties on fabric characteristics.

Figure 5 shows the effect of the processing operations on the fiber and fabric breaking elongation. The six cottons are again divided into three groups of two: a high breaking elongation group (Pima and Lengupa), an intermediate breaking elongation group (Acala and Sea Island), and a low breaking elongation group (Hybrid and Deltapine). Very clearly, the processing operations tend to reduce the differences in fiber breaking elongation among the three cotton groups in the bale stage. The rate of decrease of fiber breaking elongation due to processing is much larger for the high elongation group than for the intermediate elongation group; whereas the fiber breaking elongation does not significantly decrease for the low elongation group due to processing through mercerization. Again the effect of resin-finishing is to decrease the fiber breaking elongation the same extent for all three cotton groups. The fabric breaking elongation, also indicated in Figure 5, follows roughly the same pattern as the fiber breaking elongation. The relative positions of the three cotton groups is the same for fabric as for fiber breaking elongation. A comparatively small decrease in fabric breaking elongation is observed upon bleaching and mercerizing, while a large decrease in fabric breaking elongation is observed upon resin-finishing. The important point to note is that the same pattern was seen in the fiber breaking elongation. Just as the differences in fiber breaking elongation among the three cotton groups are to a large measure eradicated by processing, so are the differences in fabric breaking elongation among the cottons in the scoured condition greatly reduced by the time the resin-finished fabric is considered. Thus, once again on the basis of elongation properties, a good indication is available of the influence of fiber properties on fabric performance.

Figure 6 shows the effect of processing on the fiber elastic modulus. The cottons are again divided into three groups: a high modulus cotton (Hybrid), an intermediate modulus cotton group (Pima and Sea Island) and a low modulus group (Deltapine, Acala and Lengupa). The fiber elastic modulus is consistently increased by processing steps up to spinning, is increased by bleaching and mercerizing, and is decreased by resin-finishing. The basis for

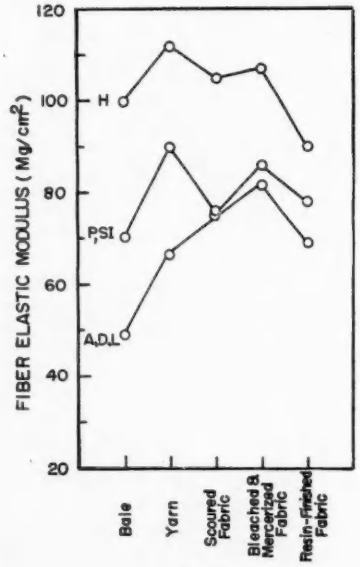

Fig. 6

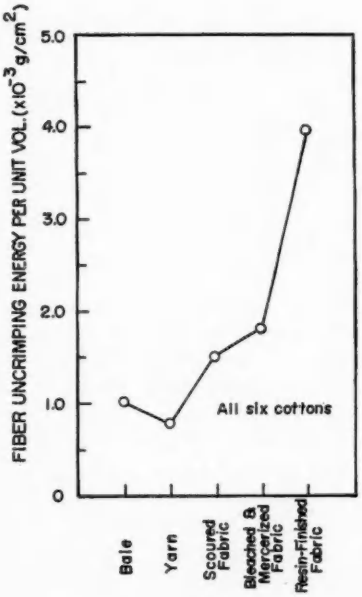

Fig. 7

Fig. 6. Effect of processing on fiber elastic modulus. H—Hybrid, P—Pima, SI—Sea Island, A—Acala, D—Delta, L—Lengupa.

Fig. 7. Effect of processing on fiber uncrimping energy.

the irregularity of these curves can be hypothesized only at this time. Conceivably, alternate steps in processing increase the fiber elastic modulus due to tensioning. The other steps are performed on fibers in the wet state, where the fibers are allowed to relax and thus decrease their elastic modulus or stiffness. The important point to note is that despite the fluctuations the relative positions of the three cotton groups are maintained throughout processing although the differences in fiber elastic modulus in the bale stage are greatly reduced by the time the fibers from the resin-finished fabric are under consideration.

Figure 7 presents the effect of processing on the fiber uncrimping energy. In this property the six cottons could not be successfully differentiated and they are therefore considered as one group. The effect of spinning is to decrease the fiber uncrimping energy. Weaving introduces fabric crimp, thus considerably increasing the total fiber uncrimping energy. Bleaching and mercerizing further increase the fiber uncrimping energy but the largest increase in this property is due to resin-finishing. As previously discussed, this increase in uncrimping energy on resin-finishing indicates a surface deposition of a portion of the resin rather than a complete penetration of the finishing agent into the core of the fiber.

Conclusions

On the basis of this research several important conclusions can be reached.

1. All processing steps exert an influence on the mechanical properties of cotton fibers. Some of these processing steps have a more radical effect than others on fiber breaking stress and elongation, fiber elastic modulus, and fiber uncrimping energy. The wet-finishing operations, such as bleaching, mercerizing, and resin-finishing, generally alter the fiber properties more than such mechanical operations as carding, spinning, and weaving.

2. The relative magnitude of the changes of fiber properties due to some treatments is not constant for all cottons, but is dependent upon the value of the original fiber property. This effect has been shown for bleaching and mercerizing. Resin-finishing, on the other hand, exerts a constant effect (particularly decreases in fiber breaking stress and elongation) on all the cottons considered.

3. In many cases the changes in fiber properties due to a given treatment are reflected in the change of the fabric characteristic. This fact is considered good evidence for the premise that cotton fiber properties influence the behavior of the resultant fabrics.

Acknowledgment

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Studies on Cotton Opening and Cleaning

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Introduction

The Whitin Axi-Flo cleaner was introduced to the industry in mid 1954 together with the Axi-Feed opening unit, which at that time was a separate accessory applied to the Blending Feeder. The Axi-Flo cleaner is a relatively simple machine having two pin-covered cylinders rotating in the same direction and parallel to the input-output flow line of the fibers processed. Fibers pass around these cylinders with their specially designed pins and are exhausted to a cage section and thence to a succeeding cleaner such as a Buckley Downstroke, or for high grade cotton (Middling or better) directly to the Picker.

The distinguishing characteristic of the Axi-Flo is that the very effective cleaning action takes place as a result of buffeting agitation of the small tufts delivered by the Axi-Feed as against the conventional and long used feed roll and beater principle.

Test results detailed later will show performance characteristics of the Axi-Flo. In addition to its cleaning capacities, the machine has demonstrated excellent blending performance for synthetics and blends, indicating a broader field of application and versatility.

The Axi-Flo has been on the market for 2½ years and has been very successfully installed in mills in the United States, Canada, many South American countries, and in other foreign countries. Some 150 of these mills are processing virtually every grade and type of cotton from low grade mixes of cotton, noils, and card strips to the finest long staple for up to 150s.

This is indeed a unique and unusual record for a new textile machine and extremely significant for a new machine in the opening line. Mills are so closely dependent upon their opening room equipment, especially if there are only two lines, that they adopt new type opening and cleaning machinery only when it is proven successful. The penalty for being wrong is grievous.

In each of the mills in which these Axi-Flo and Axi-Feed units were installed, the existing opening, cleaning, and picking machinery layouts were examined carefully. The results after the new machines were installed were also explored thoroughly. A unique opportunity was provided to explore the use of many, if not virtually all, types of machinery and combinations of cleaning machinery now used. A wide range of practices were analyzed and evaluated. Some were good, many were indifferent, and some were poor.

The data and experience provided a fairly broad base for knowing what mills are doing in their opening rooms today, what equipment is being used, and deficiencies in practice. They also provided Whitin with new higher objectives toward which to move, provocative challenges, and inspiration for future improvements.

These questions could be fairly asked. What should efficient opening and cleaning machinery do? How close are we to doing it with our present machinery? The answer to both is subject to discussion and divided opinion, but is offered here as based on present knowledge and experience.

Ideally speaking of cotton of course, efficient opening and cleaning machinery should do the following: remove all the foreign matter completely: leaf, twigs, grass, bolls, dust, dirt, everything; remove all undesired elements of cotton present, such as neps and motes and the shorter fibers. Conversely, the cotton should be manipulated as little as possible in the fewest number of machines, the long fibers left intact, and it should not be curled or affected in character.

Now going to the second question, as to our success in achieving that ideal. If we take industry's present practice as our gauge, the performance is far short of the goal. There is substantial evidence that in many mills the cotton leaves the opening room in worse condition in some ways than when it went in. Some of the dirt and undesirable matter has been extracted to be sure, but too much of the remainder has been pulverized into peppery dust inextricably attached to individual fibers, many long fibers broken, reducing its spinnability and value as well as being curled, overbeaten, and neppy.

[1] Assistant director of research.

The advantages of good opening and cleaning are clear, but often overlooked in mills whose attention is focussed on larger operations involving great numbers of more expensive machinery. The better the cotton is opened, the better the mill runs. Pickers produce better laps and the cards and all the machinery in the mill run cleaner and more efficiently, the yarn is evener and stronger. Proof is available that fabric not only has increased strength, but in some instances is improved in appearance and color.

Accordingly, we would like to discuss some of the common practices now used in opening rooms and the unfavorable results of their use. Test data will show how those difficulties can be eliminated and the contributions made by the Whitin Axi- Flo and Axi-Feed toward improvement in this area.

Blending and Mixing

Blending Feeders are the accepted and suitable machines for blending and mixing the cotton fibers. Opinions differ widely, however, in regard to the optimum number of bales which should be used for satisfactory blending. One practical limiting factor is the capacity of the bin of the Blending Feeder, and another, the smallest amount of cotton the tender can lift or strip from each bale. Since the smallest pieces of cotton the tender takes off the bale is in the neighborhood of 10 lb. and the capacity of the bin of the Blending Feeder is approximately 60 lb., it is obvious that about 6 bales per Blending Feeder is the optimum practical figure.

The purpose of blending is to average out the differences in quality between bales of cotton so that the blended cotton is uniform during the spinning process. A mixing program should be set up to even out variation between lots of bales.

To maintain an even blend, the Micronaire device is undoubtedly helpful. Its use results in maintaining the reasonable average in fiber fineness which is necessary for stable spinning conditions.

A further move in the right direction is the often neglected practice of opening the bales and allowing the cotton to bloom and become stabilized to the humidity and temperature conditions prevailing. A 24-hr. period should be allowed.

Overloading the Blending Feeders

It is quite often found that the number of Blending Feeders is insufficient, usually because of inadequate space. They are frequently overloaded and forced to run up to 300–350 lb./hr., instead of the recommended 160–200 lb./hr.

To explore the effect of overloading the Blending Feeders on cleaning efficiency, we processed SLM 1 $\frac{1}{16}$ in. cotton through our opening and picking line in our Research Department. This line consists of the following: Blending Feeders with Axi-Feed, Feed Table, followed by the Axi-Flo and Downstroke Openers and Cleaners, and a short 20 ft. duct feeding to a two-beater Picker. The result of this overloading test is shown in Table I.

TABLE I. Overloading, Blending Feeders

Production rate	Line cleaning efficiency, %
Blending feeders 300 lb./hr.	60.5
Blending feeders 180 lb./hr.	65.5

Good preparation helps the cleaning and the evenness too; hence, we always recommend the use of two Blending Feeders per Picker.

It is not very popular here in the United States, but many in the textile industry abroad are convinced that the best way to assure an even feed to the individual Blending Feeder is by the use of an extended feed apron. The apron drive is controlled by the swinging door in the bin of the Blending Feeder and when this is properly set, chokes and machine breakdowns are infrequent or avoided entirely. This type of apron helps to secure a better blend. They require considerable floor space and that is their distinct disadvantage. Further, best results are secured when the Blending feeders are set to their lowest rate of feed, and the cotton level in the bin of the first Hopper Feeder of the Picker line will fluctuate less. In practice, this has a beneficial effect on lap evenness and even on nep count, as shown later in experimental test results.

Long Ducts

In a modern spinning mill layout, the warehouse, the room for the conditioning of the bales, and the rooms for the blending, opening, and picking machinery should be adjacent. This means reduction in transportation costs and short ducting between the various machines. Mills having this type of layout

benefit in cleaning, lap evenness and for a low nep count.

Some mills are not so fortunate. Often, the mixing, or the mixing and opening room, is some distance away from the picking machinery. Experiments have shown that long ducts with the dust trunks produce an increase in tuft weight, counteracting what was achieved in the opening line. Sometimes the distances are so great that the cotton has to be drawn through a fan. This is, of course, an even worse situation and detrimental to the nep count.

A specific test was conducted in our Research Department with SLM cotton where, in one instance, the shortest possible ducting was used between the opening and picking machines and a paddle-type separator was used to avoid any rolling of the stock. In the other test, a long duct was used and the cotton was drawn through a fan. The difference of 7.4% in nep count is shown in Table II.

TABLE II. Duct Length and Nep Count

Machinery and ducting	Nep count per 10 grains
Short pipe used	270
Long pipe and fan used	290

Compressing the Cotton during the Opening Process

Repeated tests showed that maximum opening can be obtained when the cotton is loosened gradually during the opening process. Also, compression by feed rolls or by other means, as often happens on conventional machinery, should be avoided. On the present Whitin opening and cleaning line the stock is always kept in a loose, floating state from the Blending Feeder through the Axi-Feed, Mixing Feed Table, and Axi-Flo. In recent tests, resort has been made to the "paddle" type condenser to eliminate the rolling action of the cage type condenser, an effect which is often noticed in mill operation.

Figure 1 shows the view of the "paddle" type Air-Flo separator as it was used over the Hopper Feeder of the picker line.

Figure 2 shows the general design with the entrance pipe, the perforated sheet sides for separation of the stock, and the paddle wheel. This type of

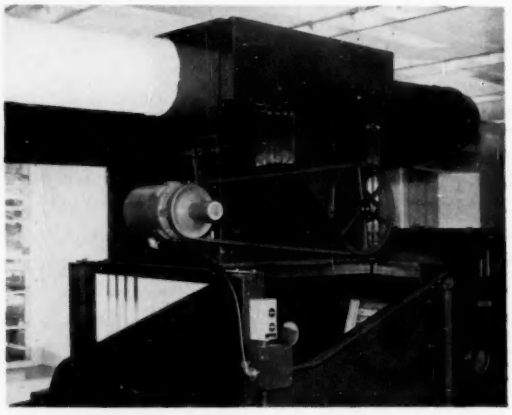

Fig. 1. Whitin "paddle" type Air-Flo separation.

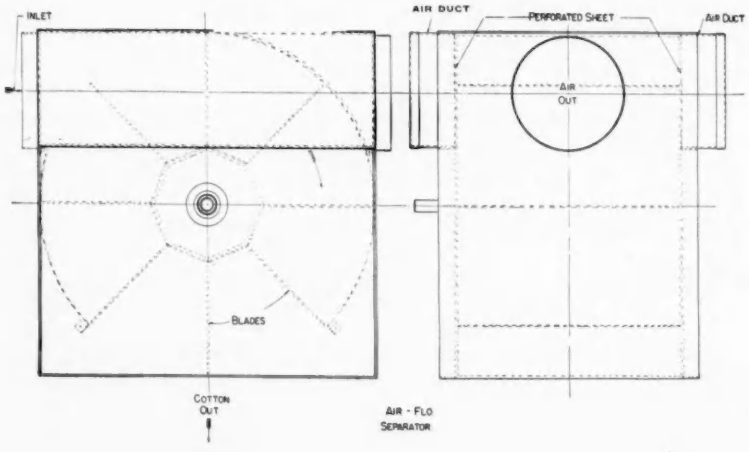

Fig. 2. General design of Air-Flo separator.

separator delivers the cotton in an open state. On an opening line used for cotton graded middling or better, the stock is never compressed until it arrives at the feed rolls of the breaker section of the Picker.

Insufficient Opening and Cleaning

Looking over the opening rooms of the different spinning mills, a wide variety of machines is found in use. Not only does the type of machinery vary markedly, but the number of different opening machines varies also within wide limits.

There are mills where the opening line is short or even nonexistent, leaving the cleaning to the picker and the card room. These installations might work when using high grade, clean cotton, but it is by no means an efficient procedure. For best over-all results, cleaning should be done in the opening room prior to the picking.

To demonstrate the effect of good opening and cleaning on nep count, card waste and yarn properties, a number of interesting tests were made using three different machine organizations.

In organization A no opening machinery was used and the cotton was fed from the Blending Feeders to the two beater Picker.

With additional machinery to show the effects of overmachining in organization B we used: Blending Feeders, three Porcupine type Downstroke Cleaners, followed by the same two beater Picker.

Organization C included the present Whitin line consisting of Blending Feeders, Axi-Feed, Feed Table, Axi-Flo, Downstroke Openers followed by the two beater Picker. The Downstroke was bypassed for the SM cotton.

Strict Middling $1\frac{1}{32}$ in. staple cotton was used first and nep count was made on the card web; of course, the same card, roving, and spinning organization and settings were used during the entire test. We found that when the opening machines are eliminated the nep count increased by 10–13%, and the amount of flat strip and lickerin wastes increased, as shown in Table III.

By eliminating the opening machines, the major portion of the motes remained in the stock, being opened on the card and causing an increase in the nep count. Since in that instance, all cleaning was performed by the picker and card, it was obvious that the amount of lickerin waste and flat strip must increase.

TABLE III. Effects of Eliminating Opening Machinery on Nep Count and Card Waste

Test No. 1, SM cotton

Machinery organization	Nep count per 10 grains	Lickerin waste	Flat strip
A no opening	340–350	Incr. by 8.2%	Incr. by 6.5%
C present line	310–315		

TABLE IV. Effects of Eliminating Opening Machinery on Yarn

Test No. 1, SM cotton

Yarn properties	Org. A.	Org. C.
Avg. unevenness	114.4	103.1
Deviation, %	20.1	15.2
Size variation, %	4.0	1.7
Break factor	2205	2285
Grain break	301.5	307.2

TABLE V. Effects of Eliminating Opening and Cleaning Machinery on Nep Count and Card Waste

Test No. 2, SLM cotton

Machinery organization	Nep count per 10 grains	Lickerin waste	Flat strip
A no opening	310	Incr. 36%	4.8%
C present line	275–270		

After spinning, the yarn was tested and the results are shown in Table IV.

In every instance, the yarn prepared on organization C was better, including appearance of the yarn on the blackboards (Figure 3).

For the first test, as mentioned above, we used a good grade cotton. Then, we repeated the test with an SLM grade, $1\frac{1}{16}$ in. staple.

The card waste and nep count test results are shown in Table V.

The nep count increased, as it did with SM, by 11–13%. Because of the higher non-lint content of the SLM, the increase in the lickerin waste is naturally very much higher than it was in the test using the high grade cotton.

The mechanical properties of the yarn spun from the $\frac{11}{16}$ in. staple SLM cotton are shown in Table VI.

As in the previous test, the yarn from organization C indicates better characteristics for every item.

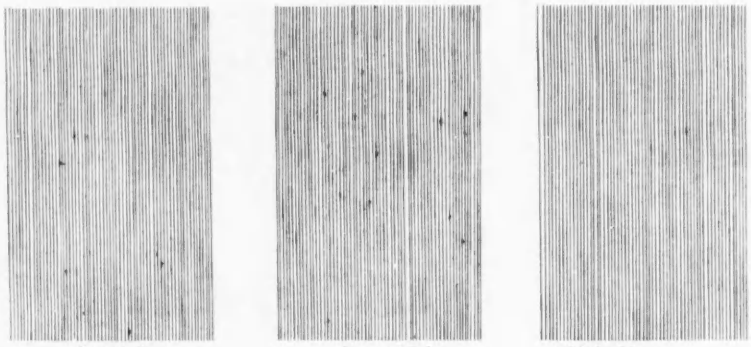

Fig. 3. Yarn appearance, shows effect of eliminating opening machinery and effect of overmachining.

TABLE VI. Effects of Eliminating Opening and Cleaning Machinery on Yarn

Test No. 2, SLM cotton

Yarn properties	Org. A.	Org. C.
Avg. unevenness	101.2	94.0
Deviation, %	19.3	17.0
Size variation, %	4.9	3.5
Break factor	2190	2350
Grain break	291.6	312

In an earlier test, we illustrated the effect of opening on the regularity of the picker lap. For these series of tests, since we were not interested in the cleaning, we used 1½ in. staple 1.5-den. viscose staple. First, we processed the material through a complete line comprised of Blending Feeders, Axi-Feed, Axi-Flo, and a two beater Picker, similar to the machinery used in the previous organization C. (Downstroke by-passed).

As a next step, the Axi-Flo opener was by-passed and staple fiber went from the Axi-Feed to the Picker. Finally all opening machines were by-passed and the stock was fed from the Blending Feeders straight to the Pickers, as in organization A, mentioned above. The laps were checked on the Uster Tester and the resulting diagrams are shown in Figure 4.

The top diagram indicates a very even lap from the complete line. The second diagram shows a lap that is not even, due to the lesser degree of opening. The third graph shows the most uneven lap. For this lap, all opening machines were by-passed. Due to lack of adequate opening, the tufts in the lap are bigger, affecting the inch-by-inch evenness unfavorably.

Overbeating

It is well-known that machine-picked cotton creates serious problems in opening rooms. Conventional or older type machinery simply cannot do really effective cleaning. In an attempt to solve the problem, it has been common practice for mills to add cleaning machines to their lines. Quite frequently opening machines were used in tandem.

Concerning the tandem use of opening machines, tests done by the Shirley Institute in 1935 showed that this practice was relatively ineffective. Our own tests confirm these results. Hence we do not recommend the use of machines in tandem.

If for any reason two identical opening machines must be used in the opening line, the best procedure is to insert a different type of machine between them.

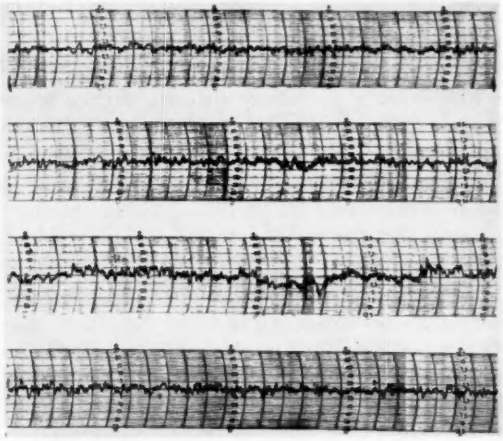

Fig. 4. Uster lap-test charts.

If only a hopper feeder is used as the intermediate machine, it will secure a rearrangement and redistribution of the cotton tufts, a factor helpful in the cleaning process.

In exploration of this subject and to determine the effect of the use of more than sufficient machinery and its relationship to nep count and yarn characteristics further tests were made with the same SM and SLM cotton qualities.

In test No. 3, the cotton was processed on organization B through the Blending Feeders, then passed through a Downstroke Opener three times and thence to a two beater Picker. This test reveals very clearly and underscores the widely held opinion that overmachining increases the nep count. The results of this test are shown in Table VII.

The effects of overmachining on the spun yarn is shown in Table VIII. Here again a comparison is made, processing SM cotton on the machinery organizations B and C.

Tables IX and X show the effect of overmachining on the SLM cotton.

In the tests data above, it appears clear that the use of minimum number of effective opening and cleaning machinery gives better results. It should also be kept in mind that to keep the nep count to a minimum, the multiple use of the same type of machine should be avoided, especially in tandem.

These research tests confirm in the laboratory the results secured in mill practice from the Whitin Axi-Feed and Whitin Axi-Flo. In general, both mill and laboratory tests show that on the average a nep reduction of at least 10%, and in some instances as high as 30%, should be secured over what can be done with conventional opening equipment.

The importance of all of these tests does not lie so much in the actual figures shown, because of wide variation in cotton quality and the types and arrangements of machinery used in mill practice. The distinctive and outstanding fact to be noted is the definite indication that nep count increases and the quality of the yarn decreases whenever the opening is insufficient or when too many machines are used or identical machines used in tandem.

In other words, the minimum nep count and the best yarn quality are secured when opening and cleaning is done in a properly equipped opening room and when good practices are followed to avoid long ducting.

Now turning to the future, the question can easily be asked: What progress can be expected? Going back to our definition of ideal opening and cleaning, we pointed out that all of the foreign matter such as neps and motes should be removed, and that this should be done without damage to the cotton quality or character. Both sections of this definition, the positive and the negative, are extremely important.

It seems now we can say with surety that substantial progress has been made toward the first of the objectives, the removal of all of the foreign

TABLE VII. Effects of Overmachining on Nep Count and Card Waste

Test No. 3, SM cotton

Machine organization	Nep count per 10 grains	Lickerin waste	Flat strip
B overmachining	355–360		Incr. 3%
C present line	310–315		

TABLE VIII. Effects of Overmachining on Yarn

Test No. 3, SM cotton

Yarn properties	Org. B.	Org. C.
Avg. unevenness	108.1	103.1
Variation, %	18.7	15.2
Size variation, %	3.7	1.7
Break factor	2225	2285
Grain break	304.8	307.2

TABLE IX. Effects of Overmachining on Nep Count

Test No. 4, SLM cotton

Machine organization	Nep count per 10 grains	Lickerin waste	Flat strip
B overmachining	290	Incr. 13.6%	Incr. 2.5%
C present line	275–270		

TABLE X. Effects of Overmachining on Yarn

Test No. 4, SLM cotton

Yarn properties	Org. B.	Org. C.
Avg. unevenness	100.8	97.0
Deviation, %	18.3	17.0
Size variation, %	4.8	3.5
Break factor	2240	2350
Grain factor	308.4	312

matter and undesired cotton material in the mix. As a general statement it may be said that acceptable mill standards now are met by the removal of 50% of this waste material by the time the cotton is in the picker lap, with a few mills doing slightly better than that. With the Whitin Axi-Feed and Axi-Flo and utilizing our present knowledge of machinery layout and number of machines, we feel that mills should expect to, and should, remove from 65 to 68% of this material.

As reported above, this can be done and is being done in many mills with a reduction of 10 to 30% in their nep count as compared to their former results, indicating that progress is being made in that respect also. We do expect further progress in both of these areas. It seems that within the foreseeable future that an even greater amount of waste can be extracted successfully, while retaining the advantage of a lowered nep count and avoiding all of the damage done to cotton by overbeating and overmachining. Prophesy is dangerous but enticing. A reasonable goal now is to remove 80–85% of the waste by the time the cotton is in the picker lap and perhaps to reduce the nep count by another 5% from the low levels now secured.

Even now, the principles and general methods for securing these objectives are clear. Specifically, we are thinking in terms of using Blending Feeders with Axi-Feeds, one Axi-Flo if good grade cotton is processed, followed by another cleaning unit if lower grade cottons are processed. The line will feed to a more efficient two-beater picker than the conventional one now being used.

To reach these goals, we are doing an immense amount of study and research and translating the knowledge gained into improved machinery.

The Relation of Twist to the Construction and Strength of Cotton Rovings and Yarns[1]

C. B. Landstreet,[2] P. R. Ewald,[3] and J. Simpson[4]

Introduction

The strength of roving and yarn when other factors are constant, is a function of twist. While rovings are not twisted for maximum strength, the twist is nevertheless important. Under-twisted roving causes breaks in the creel and stretching while over-twisted roving drafts poorly and is time consuming in its production.

If yarns are to exhibit maximum strength, correct twist is absolutely necessary. While it is true that for most yarns the twist-strength curve is rather flat in the vicinity of maximum strength, it is desirable to know at just what twist rate maximum strength occurs. Spinning beyond this point is again time consuming and alters other yarn characteristics. Spinning before this point greatly increases the chance of weak yarn due to the asymmetrical shape of the twist-strength curve.

In many instances it is desirable to be able to predict accurately from data on a particular yarn number the twist to produce maximum strength in other numbers made from the same material. An equation for doing this will be developed here which has been used successfully over a number range from 4/1 to 100/1 yarn.

Another problem that arises in yarn manufacturing is that of producing a yarn of a given number and twist. To do this successfully requires a knowledge of the relation between the five factors: original yarn number, final yarn number, yarn contraction, yarn

[1] Contribution from the Spinning Laboratory, Crops Research Division, A.R.S., U.S.D.A. and the Tennessee Agricultural Experiment Station, Knoxville, Tennessee cooperating, and the Southern Utilization Research Branch, New Orleans, Louisiana, presented at the Cotton Research Clinic, Savannah, Georgia, Feb. 28, 1957.

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[3] Engineer, Spinning Laboratory, U.S. Cotton Field Station, Knoxville, Tennessee.

[4] Cotton Technologist, Southern Utilization Research Branch, New Orleans, Louisiana.

twist, and machine twist. An equation will be developed here combining the necessary factors involved in making a given yarn from a particular cotton.

Recent studies have been made on roving twist and strength by Simpson [1, 6], Smith [7] and others and on yarn twist by Fiori [3], Sustman [9], Heiberg [4], and Duerst [2].

Roving Twist

Work on roving twist has been facilitated by the development of the roving tester shown in Figure 1. This instrument was described in a previous paper [6]. The range in strength that can be tested is from about 5 to 500 g. In Figure 2 are shown twist-strength curves for seven rovings made from carded Deltapine, $1\frac{1}{16}$ in. cotton. These curves show the effect of number from 0.50 to 10.0 on the twist-strength curve. Along with number the positions are

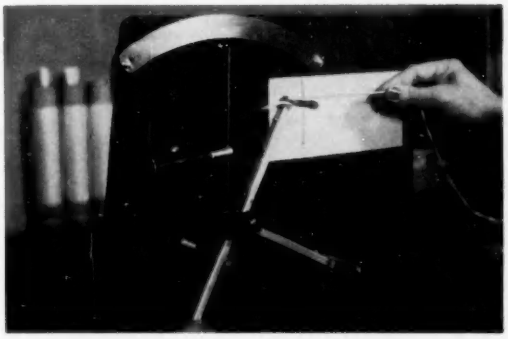

Fig. 1. The roving strength tester.

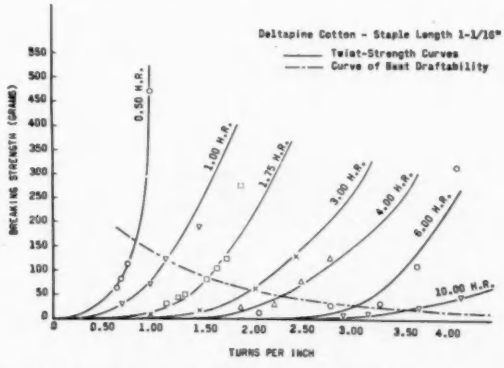

Fig. 2. Twist-strength curves for seven rovings tested on the roving strength tester.

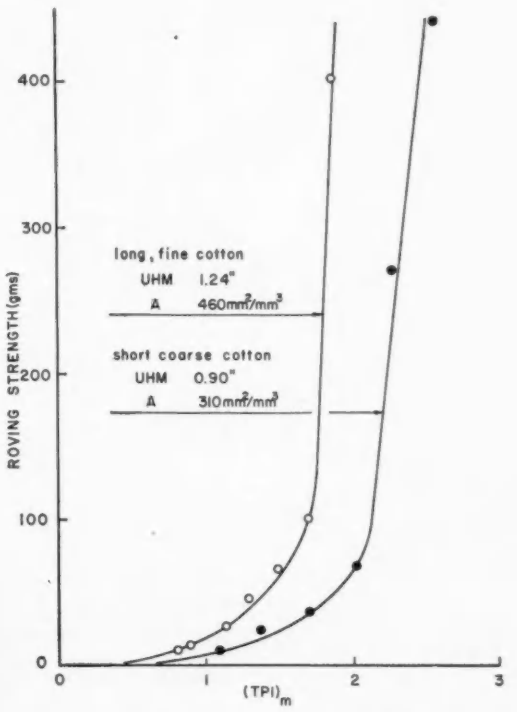

Fig. 3. Twist-strength curves showing the effect of fiber properties on the position of the curves for 2.0 hank roving.

governed by many factors, among which are fiber properties, roving age, position on the bobbin, and method of manufacture. In Figure 3 are shown the twist-strength curves for 2.0 hank roving made from cottons differing widely in fiber properties. The longer, finer fiber requires less twist to produce a given strength than does the shorter, coarser fiber.

Although there have been many studies of the twist strength curve in the area of maximum strength, little has been done experimentally in determining the shape of the over-all curve. For instance, it was not known whether the curve beyond the point of maximum strength keeps bending in a convex manner and intersects the abscissa or whether it had a point of inflection and approached the abscissa asymptotically.

In an effort to determine the shape of the complete twist curve a fibrous strand was twisted by two methods. The first method consisted of taking a low twisted 4.00 hank roving of Deltapine cotton and running it through just the front rolls of the roving frame a successive number of times. Each time 4.74

turns per inch of twist were added. In this manner the twist was added in increments. The roving hank was not held constant but became coarser after each addition of twist.

A twist strength plot of the rovings made in this manner is seen in Figure 4, curve A.

The other method consisted of making 4/1 yarns with different amounts of twist on the ring spinning frame. In this case the total twist in each yarn was placed in at one time and the yarn number was held constant. A twist strength plot of these yarns is shown in Figure 4, curve B. It can be seen that both curves have points of inflection beyond the maximum point.

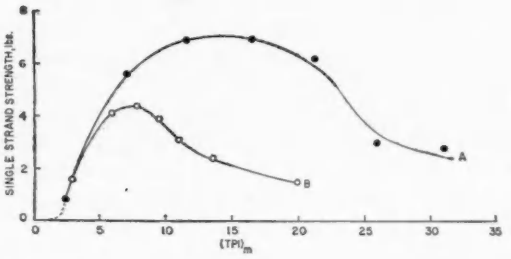

Fig. 4. Twist-strength curves developed over a wide range in twist.

Yarn Twist

Previous Work

The part of the twist-strength curve that has received the most attention is that in the area of maximum strength. It has been shown theoretically by Sullivan [8] that the twist multiplier to produce maximum yarn strength is a function of the product of fiber friction, length, and specific area. Recently the authors, using Sullivan's work as a guide developed and published a working twist guide [5]. At that time the twist multiplier was defined simply as

$$TM = \frac{(TPI)_m}{\sqrt{\overset{*}{C}}}$$

The turns per inch was defined as the twist constant divided by the twist gear, or what will be called here mechanical twist, $(TPI)_m$. Later experiments have indicated that this equation will not give desired results for a wide range in yarn numbers. This has been pointed out before by Duerst [2], Heiberg [4], and others. In the authors' original work no allowance was made for yarn contraction due to twist and as a result the final yarn always had more twist in it than given by this twist equation.

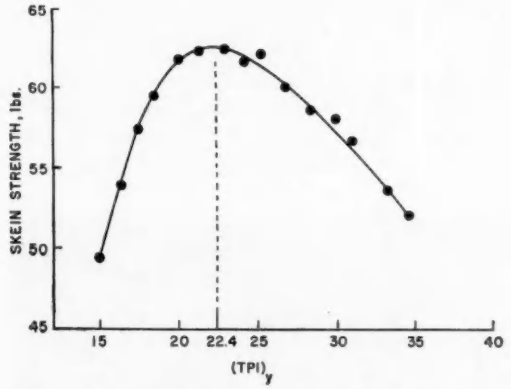

Fig. 5. A representative twist-strength curve for yarn, in the area of maximum strength.

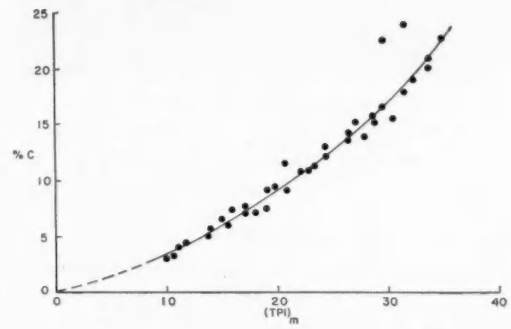

Fig. 6. A typical twist-contraction curve.

Development of New Twist Factor J

Twist-strength curves similar to that shown in Figure 5 for selected yarn numbers from 10/1 to 100/1 and 10/1 to 60/1 were run respectively for two cottons differing widely in fiber properties. Contraction curves, one of which is shown in Figure 6, were developed for each number. The yarn contraction is defined here as:

$$\% C = \frac{N_o - N_f}{N_o} \cdot 100 \qquad (1)$$

where N_o is the original number, or the number at zero twist, and N_f is the final yarn number. Using

the contraction curve, the turns per inch in the yarn, $(TPI)_y$, was calculated as follows for each experimental point on all twist strength curves:

$$(TPI)_y = \frac{(TPI)_m}{1 - C'} \qquad (2)$$

where C' is the length decrease per inch of delivered yarn length. Thus the abscissa of the graph in Figure 5 represents the actual twist in the finished yarn. The point of maximum strength for each number was selected by inspection from the twist-strength curve and those points have been plotted as shown in Figure 7, curve A and B. Curve C shows the error that would exist if the square root equation had been applied starting at 10/1 yarn on Curve B. Log versus log plots of curves A and B are shown in Figure 8. It can be seen that the slopes are almost identical even though the cottons vary greatly in fiber properties. For this reason it has been assumed that fiber properties are of second order importance, and an average slope of 0.54 has been selected. The twist equation thus becomes

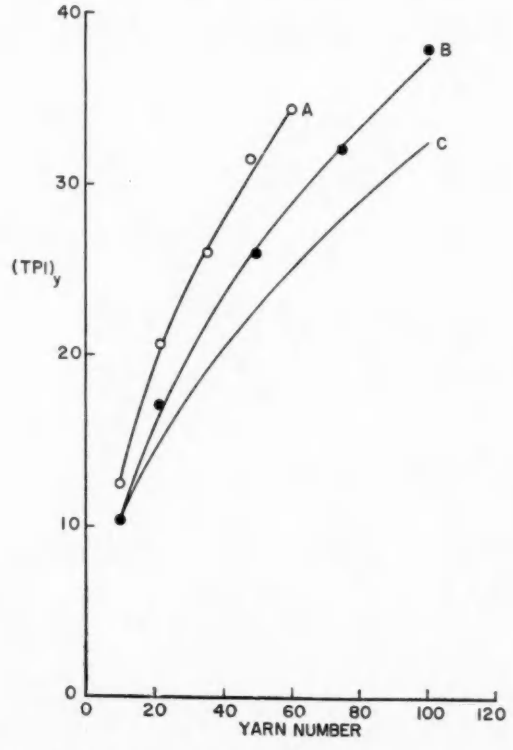

Fig. 7. Curves showing the relation between yarn number and twist.

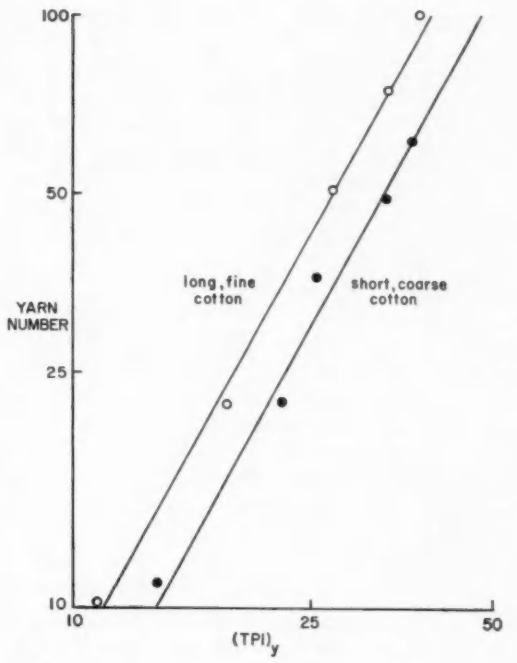

Fig. 8. Log versus log plots showing the relation between yarn number and twist.

$$(TPI)_y = N_f^{.54} \cdot J \qquad (3)$$

where $(TPI)_y$ is the twist in the yarn, N_f the final yarn number, and J the twist factor replacing the old twist multiplier, TM. Because all the original twist work at Knoxville was performed on 22/1 yarn, it was possible to convert the old chart to the present one shown in Figure 9. The TM was converted to J, and by plotting the reciprocals of J and $M\sqrt{A}$, the approximate hyperbola was transformed into the straight line as shown. This was done for convenience only.

Development of the Number-Twist-Contraction Equation

When J has been determined from the curve in Figure 9 for a particular cotton, the resulting twist calculated from Equation 3 will be the twist in the final yarn. It is necessary to find the mechanical twist since it is used in obtaining the twist gear for

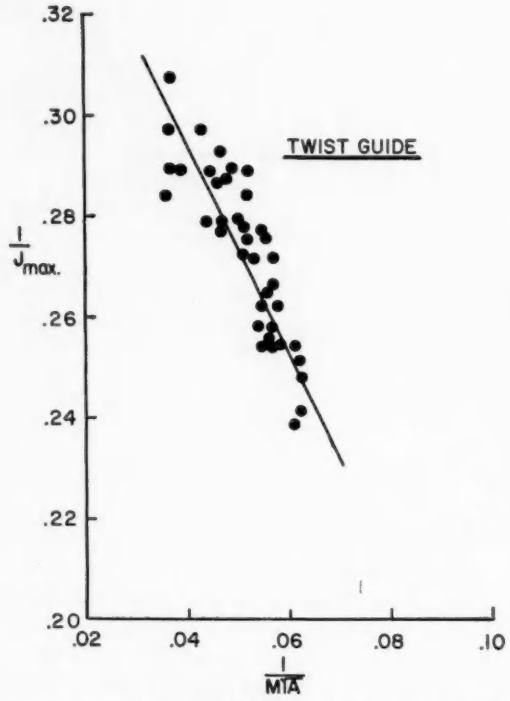

Fig. 9. The yarn twist guide used to obtain maximum skein strength in single yarns.

the spinning frame. The yarn number in Equation 3 is the number of the final yarn. To calculate the spinning draft, the original number must be found since it is this number which contracts, due to twist, to give the final yarn number. The development of equations, based on experimental data, necessary for finding $(TPI)_m$ and N_o follow.

The contraction curve shown before in Figure 6 is one of a family of curves that is formed when different numbers are twisted. The original yarn number N_o was held constant at each point, thus the contraction resulted from twisting an equal mass of material at each point. The curves have the general equation:

$$\% C = K (TPI)_m^n \qquad (4)$$

where $(TPI)_m$ is the mechanical twist. The exponent n which is constant regardless of number was evaluated by very careful experiments using an N_o of 22. This number was chosen because an extreme range in twists could conveniently be spun. A log versus log plot of the data is shown in Figure 10. The least squares line had a slope of 1.42 but it was found that in practice the line with a slope of 1.5 as shown, adequately represented the average condition.

To evaluate K, which varies with original number, a range of yarn numbers were spun at a constant twist rate and the contraction carefully measured. It was found that by selecting the proper spinning tension for each yarn number a linear relationship could be established between K and the reciprocal of the yarn number. Diagram A, Figure 11, is a plot of the experimental data showing the number-contraction curve at two different twist rates. Values for K for any original yarn number can be found by substituting the coordinates of any point on either curve in Diagram A, Figure 11, into Equation 4 and solving for K. Diagram B, Figure 11, is a plot of points obtained in this way. The equation of the line is

$$K = k \left(\frac{1}{N_o} \right) \qquad (5)$$

For the experimental data presented here, k is ap-

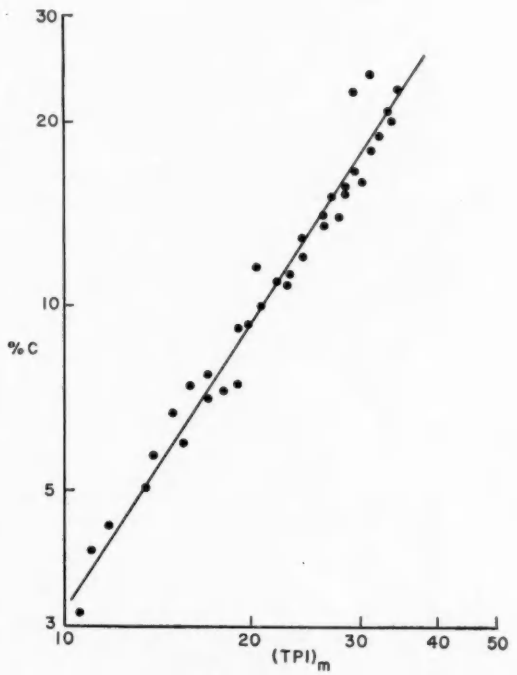

Fig. 10. A log versus log plot showing the relation between yarn contraction and twist for 22/1 yarn.

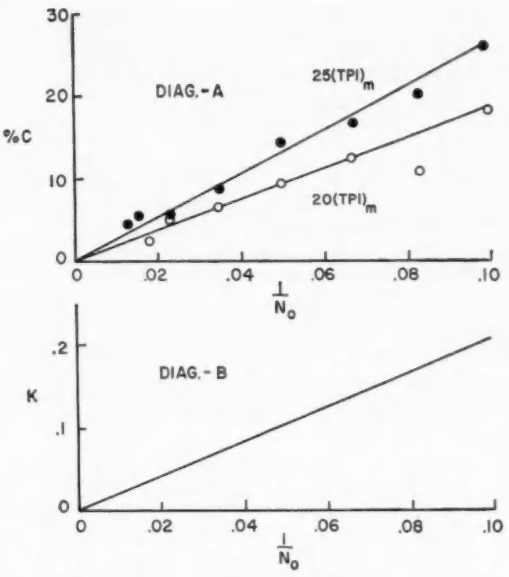

Fig. 11. Diagram A. The relation between per cent contraction and yarn number for two twist rates. Diagram B. The linear relation between K and $1/N_o$ at selected spinning tensions.

proximately 2.09. Equation 4 may be written

$$\% C = k \left(\frac{1}{N_o} \right) (TPI)_m^n \qquad (6)$$

To derive the final equation, Equations 1 and 6 are equated

$$\frac{N_o - N_f}{N_o} \cdot 100 = k \left(\frac{1}{N_o} \right) (TPI)_m^n \qquad (7)$$

Because $(TPI)_y$ will be given, $(TPI)_y \left(1 - \frac{N_o - N_f}{N_o}\right)$ must be substituted for $(TPI)_m$ giving

$$100 (N_o - N_f) = k (TPI)_y \left(1 - \frac{N_o - N_f}{N_o}\right)^n \qquad (8)$$

$$100 (N_o - N_f) = k (TPI)_y^n \frac{(N_f)^n}{(N_o)^n} \qquad (9)$$

Thus the final equation may be written for convenience as

$$N_o^{2.5} - N_o^{1.5} N_f - 0.0209 (TPI)_y^{1.5} N_f^{1.5} = 0 \qquad (10)$$

where N_o is the original yarn number, N_f is the final yarn number, and $(TPI)_y$ is the number of turns per inch in the final yarn. Equation 10 cannot be solved explicitly for N_o but a trial and error solution is not difficult.

The following example is given to illustrate the use of the Twist Guide and Equation 10. Assume that it is desired to make a 22/1 yarn from a 1.75 hank roving. Let the mean fiber length be 1.00 in. and specific surface 337 mm.^{2} per mm.^{3}. The yarn is to be spun for maximum strength. The mean length times the square root of the fineness is 18.35. From the Twist Guide, Figure 9 this gives a J value of 3.77. The $(TPI)_y$ from Equation 3 is 20.0 turns per inch in the final yarn. The above values are substituted into Equation 10 which is to be solved for N_o. From experience it is known that N_o must be larger numerically since upon contraction the yarn number will decrease. A trial and error solution gives an N_o of 23.7. Substituting this value and 22/1 into Equation 1 gives a per cent contraction of about 7.2%. Substituting 0.072 for C' and 23.7 for $(TPI)_y$ in Equation 2 gives 18.36 for $(TPI)_m$. The twist gear is then found in the conventional manner. The actual machine draft is found by dividing N_o by the hank roving. The contraction has been taken care of in finding N_o. To summarize this example: a 1.75 hank roving, drafted 13.54 (N_o equal 23.7) and turned about its axis 18.36 revolutions per inch of roller delivery will produce a 22/1 final yarn having 20 turns per inch. The yarn will exhibit maximum skein strength for that particular yarn number and cotton used.

Discussion

Roving

The strength of roving in the range of practical twists is primarily a function of roving size, twist, and fiber length and fineness. Other factors such as method of manufacture, age, and position on the roving bobbin also affect the final strength. As shown by the twist-strength curves, the roving strength increases slowly at first but when sufficient twist is reached, the rate of change is so rapid that the curve is nearly vertical. It is important to notice that the average twist used in commercial rovings is usually in the critical twist zone. As can be seen in Figure 3, at one point in the zone an increase of about two tenths of one turn of twist quadruples the roving strength. In most cases this would render the roving useless.

Work is underway to develop a better twist guide so that correct twist may be predicted from the fiber properties for any particular cotton.

Yarn

The fact that the old square root relation for calculating twist is not accurate over wide ranges in yarn numbers has been known for sometime. Various powers other than 0.50 have been suggested but have not found wide acceptance. Provided yarn density is constant, the square root relation yields constant peripheral twist angle in all yarn numbers. The variation in yarn density along with scale effect may be the explanation as to why the relation does not hold. For the average spinner the square root relation combined with mechanical twist will always be a useful tool. On the other hand, in cases such as laboratory or pilot plant work where raw stock may be too limited for general experimentation the method for ascertaining the twist for maximum yarn strength presented here should prove very useful. The new twist guide makes use of the "twist in the yarn" rather than the mechanical twist. Equation 10 combines the factors necessary for constructing any given yarn. If J is selected from Figure 9 and $(TPI)_y$ from Equation 3, the yarn will exhibit maximum strength.

Nomographs have been developed which aid in the solution of Equation 10 and they will soon be available from the Knoxville laboratory.

Acknowledgments

The authors wish to acknowledge the work of the Knoxville laboratory staff, Drs. Hertel and Craven, and Mr. Herbert Hutchens who supervised the processing of all yarns. They are also indebted to Dr. W. D. Hanson for preparing the nomographs.

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Uniformity Analysis of Yarns, Rovings, and Slivers Using Beta Radiation

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Abstract

Beta radiation is being widely applied for control purposes in the manufacture of paper, sheet rubber and plastics, and of metal foils. Its application to the manufacture of textile strands presents special problems, but in this paper it is seen that they may be resolved. A statistical type of instrument is described which makes use of the unique properties of beta radiation in studying the uniformity of cottons during processing into yarn.

Introduction

In conducting research involving the processing of cotton fibers into yarns, the need for uniformity analysis of the mass cross section of strands of slivers, rovings, and yarns is well established. A number of analyzers have been developed and placed on the market and an evaluation of these has recently been made [7].

The commercial analyzers are available with two types of detector units: pressure rollers and capacitance pickups. The pressure roll detectors will show a good relationship with mass cross section, but do not actually measure that value per se, and where fiber stiffness or fineness or strand structure may vary, there is a question as to the measurement being made. Capacitance detectors likewise make the measurement second hand, so to speak, and while serving their usual purpose quite adequately, do not make a positive measurement of mass cross section. If, however, a radiation absorption detector were to be employed, a positive indication of the mass present could be obtained. This would include the cellulose of the cotton under test and, unfortunately, the contained water as well.

To carry out the work at the U.S.D.A. Spinning Laboratory at the University of Tennessee Agricultural Experiment Station, a relatively fine structure study of the mass cross section of yarns, rovings, and slivers is needed to ascertain processing differences due to the widest known ranges of fiber properties. Because the laboratory is held at ASTM atmospheric standards of 70° F. and 65% R.H., the problem of water variability is eliminated and the radiation absorption method then offers a possibility for positive mass measurement. Accordingly, work was started to ascertain if a uniformity analyzer could be developed using a penetrating radiation for the sensing element.

[1] Crops Research Division, A.R.S., U.S. Department of Agriculture; cooperative with the University of Tennessee Agricultural Experiment Station.

Development of Analyzer

Background

The range of material to be tested, as shown in Table I, varies from 100's cotton yarn (approx. 6 Tex.) to 80 grain sliver (approx. 6,000 Tex.). The weight per unit length of the material divided by the diameter gives an approximation of the maximum absorption mass expressed in grams per cm.^{2} From the last column in Table I, it is apparent that a radiation with a range of 43 mg./cm.^{2} would pass through all the material concerned. Referring to a text on nuclear physics [1], we find that beta particles with

TABLE I. Range of Material to be Analyzed

		g./cm.	Dia., cm.	g./cm.^{2}
Yarn	100's	0.00006	0.008	0.009
	22's	0.00027	0.02	0.013
Roving	10's	0.0006	0.13	0.005
	2's	0.0030	0.25	0.012
Sliver	20 grain	0.014	1.3	0.011
	80 "	0.056	1.3	0.043

an energy of 0.3 Mev will do the job in aluminum which is close to cellulose in density. It might be well to note at this point that if a radiation is chosen which is almost completely absorbed in the maximum amount of material to be tested, there will be a minimum background or a maximum signal-noise ratio as expressed in the conventional terminology. Beta particles are thus chosen over alpha particles which are stopped by even thin paper and over gamma or X rays which are little absorbed in cotton strands and would produce an impossible background with which to work.

A number of beta-ray gauges are on the market in wide use, but an investigation showed that these are principally designed for web or sheet materials such as paper, plastics, rubber, and metal foil, and that none were applicable to yarns and rovings. Some could, however, be adapted to handle sliver or picker laps. As a consequence, it was necessary to design and construct the beta-ray gauge from the beginning.

Radiation Source

To select a radiation source, it is first necessary to consider that the source must be encapsulated to avoid contamination of the material being measured. At this point, it should be noted that the preparation of radioactive sources is no job for the amateur; in fact, the Atomic Energy Commission will sell such isotopes only to approved customers. The ease with which the radioactivity can be spread around is particularly noticeable during evaporation, pointing up the need for trained personnel in the preparation work. The encapsulating process requires that a thin window be placed over the source so that the energy requirements are raised from 0.3 Mev as noted before to about 0.7 Mev to include absorption by the window of the source and of an ion chamber. Also an allowance must be made for the distribution of beta particle energy which gives most of the particles an energy of about half the maximum value.

In looking over the Oak Ridge National Laboratory catalog of available radioactive isotopes [5], that isotope which best meets the above criteria is Thallium 204, a pure beta emitter of 0.77 Mev maximum energy and a half life of 4 years, which is a reasonable longevity considering the low cost of $5 per millicurie. A pure beta emitter is highly desirable from the standpoint of eliminating background and shielding problems.

To determine how intense a source is needed, the accuracy of the instrument must be considered. Since beta particles are emitted at random, 1000 of them must be detected before it can be said that they have been counted to an average accuracy of 2%, i.e., the per cent relative probable error equals 0.02 equals 0.67 times root N divided by N, where N is the number of particles. Assuming a strand speed of 1 cm./sec. and a resolution of 1 cm., 1000 counts per second are needed. Since only the order of 1% of the emitted particles would pass through the cotton, the source would have to give off 10^5 particles per second. Ionization chambers are close to 100% efficient, so with such a detector, 10^5 particles per second would be the maximum needed for the desired accuracy of 2% at 1 cm./sec. pulling rate and 1 cm. resolution. However, a 1 millicurie source has 3.7×10^7 disintegrations per second; hence the pulling rate might be boosted to 100 cm./sec. with another safety factor of 3.7 ×. This permits the resolution to be cut to 0.5 cm. with a 1.8 safety factor. A 2 millicurie source was selected to permit the above accuracy to be realized for the full half life of the source. Calculations showed that if the source were made 0.5 cm. long to permit maximum resolution, it could be made no narrower than 0.2 cm. in order to avoid self absorption in the source isotope itself through building too deep a layer. With these factors in mind, the source was prepared and mounted to give a radiation absorption arrangement as shown in Figure 1.

Detector-Amplifier

For the radiation detector, an atmospheric pressure, air-filled ion chamber was chosen because of its simplicity of construction. Calculations showed that with the desired range of radiation as determined above, the ion current would vary from 10^{-12} to 10^{-10}

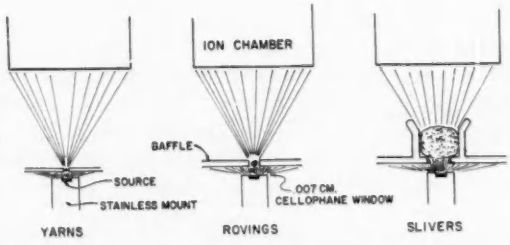

Fig. 1. Mounting and collimation of beta ray source.

amp., a range well within the capacity of the Model 145 electrometer amplifier used. A specially built preamplifier was used to match the ion chamber to the electrometer. A 0–1 milliampere d.c. strip chart recorder, plugged into the output of the electrometer, completed the equipment needed for an instantaneous analyzer.

Statistical Analyzer

The mean value is relatively easy to obtain in instrumental analysis. The instant value is run through a time constant circuit which may be set at 1, 10, or 100 sec., giving a running mean value over the last 1, 10, 100, or 1000 cm. depending on needs.

The variance, and consequently the standard deviation and coefficient of variation, is more difficult to attain in that it requires taking the difference of two large numbers which, in the instrument, requires high accuracy and stability of the circuits involved. The relationship used is the machine method for obtaining the variance which is Sum $(x^2)/N$ – Sum $(x/N)^2$. These statistical values are obtained by circuitry following the block diagram shown in Figure 2. The time constants involved must of course be equal in the mean and variance circuits for the data to be compatible.

Construction

The developmental apparatus is shown in Figure 3. The layout includes: a specially made puller, source housing, ion chamber and preamplifier; a stock electrometer; special mean circuit; special variance amplifier and a stock recorder. The squaring circuits for the variance amplifier were found in the M.I.T.

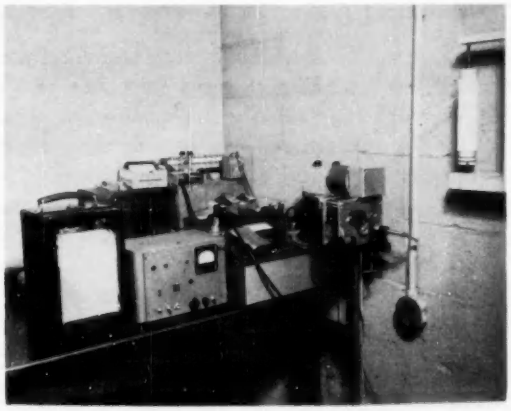

Fig. 3. Developmental apparatus.

Radiation Series [8]. D.C. amplification was used throughout with the complete circuitry shown in Figure 4. Shown in Figure 7 is a photograph (recorder not shown) of a commercial pilot model of the analyzer with some test data obtained with this model shown in Table II. There are a number of innovations in this pilot model which, however, must be the subject of another paper.

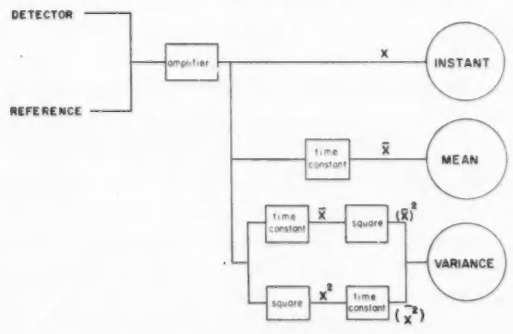

Fig. 2. Analyzer block diagram.

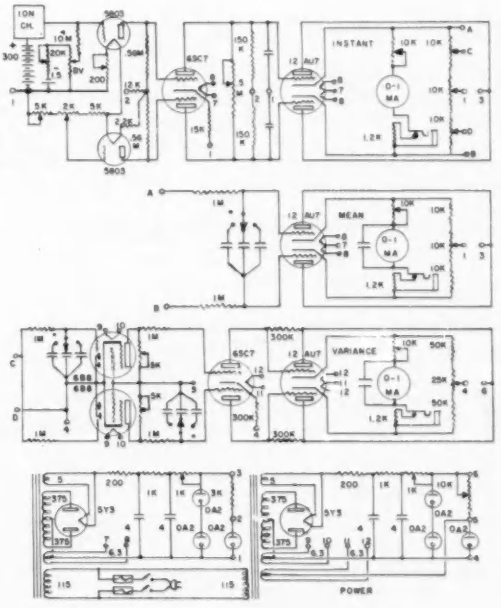

Fig. 4. Developmental circuit diagram.

Calibration and Testing

A check was made on the linearity of the analyzer by weighing a number of yarns from 70's (8.435 Tex.) through 16's (36.9 Tex.), obtaining graphs for each on the analyzer, and then plotting the mean of each graph against reciprocal count as illustrated in Figure 5, which shows the linearity to be satisfactory. Linearity is almost a necessity if the signal is to be further amplified for analysis.

As a further check on operation of the analyzer, lengths of roving were run at 2 speeds through the analyzer and the graphs carefully marked to identify check points on the roving. The roving was then cut into 1-cm. lengths and each length weighed on a milligram balance. The weighings were then superimposed on the graphs as shown in Figure 6. The agreement is seen to be good, allowing for splices where the cutting paper was joined and for power line surges.

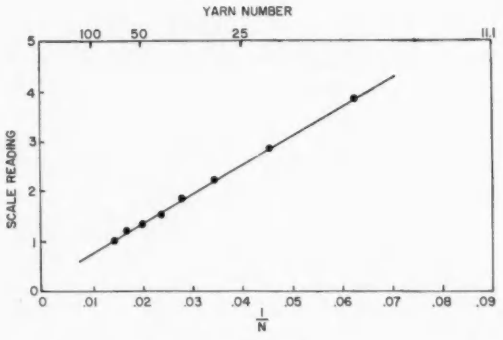

Fig. 5. Linearity of instantaneous measurement.

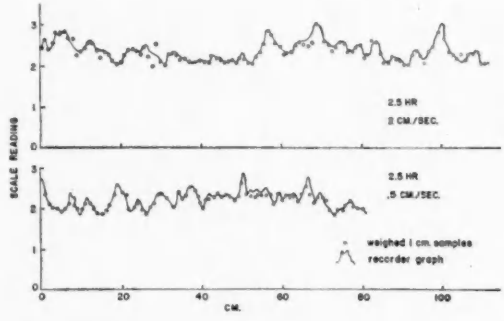

Fig. 6. Comparison of recorded and weighed values.

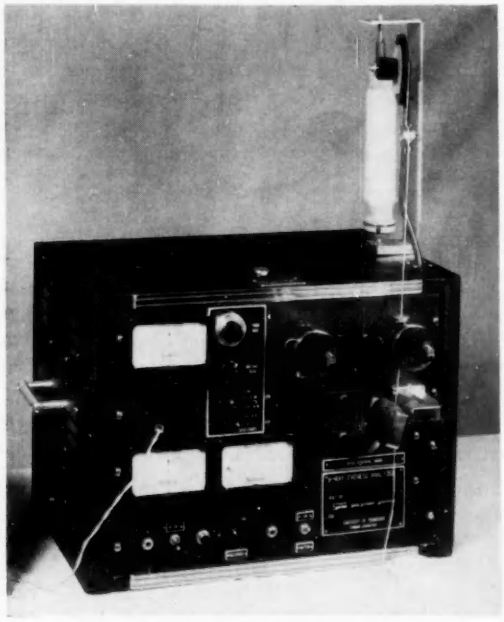

Fig. 7. Commercial pilot model of instrument.

TABLE II. Calibration of Pilot Model Analyzer

Scale Setting (Helipot)

Yarns 2.0 × Dial Reading equals Tex. (zero center)
Rovings 3.2 × Dial Reading equals Tex. (zero center)
Slivers 4.5 × Dial Reading equals Tex. (zero center)

Sensitivity

100× plus or minus 16 Tex. (zero center)
10× plus or minus 160 Tex. (zero center)
1× plus or minus 1600 Tex. (zero center)

Mean. Instant reading with 1, 10 or 100 sec. time constant.
Variance. Full scale deflection for full scale square wave signal on instant meter with 1, 10 or 100 sec. time constant.

Applications of Uniformity Analyzers

Referring to Table III, to recapitulate a comparison between existing types of analyzers and the one described, the roller detector type [2, 3, 6] measures cross-sectional area of yarns, rovings, and slivers at a predetermined weight loading; is limited in response by inertia of the rollers; and, on a segmental basis, measures instant, mean, and variance values. The capacitance detector types [4] and [3] measure the cross-sectional dielectric capacity of yarns, rovings,

TABLE III. Comparison of Principles of Analyzers

Analyzer	Detector	Pulling rate cm./sec.		Type of analysis: Instant 0th	Mean 1st	Variance 2nd	Skew dist. 3rd	Symmetry dist. 4th
Pacific [2, 3, 6]	Rollers	18	Segmental	x	x	x		
Uster [4]	Capacitance	153 max.	Continuous	x	x	(A)		
Brush [3]	Capacitance	153 max.	Continuous	x	x	(B)		
U.-Tenn.	Radiation	1, 10, (100*)	Continuous	x	x	x		

(A) Measure differential summation, related to variance.
(B) Measure deviation summation, related to variance.
* Possible rate, not used in present analyzer.

and slivers; are limited in response by the size of capacitance plates used; and, on a continuous basis, measure instant, mean and differential or deviation values which may be related to variance. The radiation detector type measures the cross-sectional mass of yarns, rovings and slivers; is limited in response by the statistical fluctuation of the source; and, on a continuous basis, measures instant, mean, and variance values.

The question of which is more desirable, segmental or continuous analysis, is a matter of choice. A segmental analysis should be the same regardless of which direction the strand is run. A continuous analysis will be influenced to a progressively lesser degree, depending on the time constant used, by strand material which has gone before. In either case, of course, the more material that is analyzed, the less variable will be the data.

Note that all the analyzers measure, to some degree, the three statistical moments; 0th, 1st and 2nd. If the variability measured is normally distributed, the uniformity is completely described by these moments. If, however, there is skewness in the distribution, i.e. a 3rd moment is present, or if there is a different peakedness of distribution from normal, i.e., the 4th moment is different from normal, then the analyzers still have not given a complete story. The skew distribution could well be introduced by nonlinearity in the processing equipment, and the symmetrical or variation from normal peakedness by instability. The automatic attainment of 3rd and 4th moment measurements is a job of some difficulty however, judging from the effort necessary to attain the 2nd.

Acknowledgments

The authors wish to acknowledge the helpful comments and advice of Dr. K. L. Hertel and Dr. G. K. Schweitzer of the University of Tennessee and of Mr. Hugh Neil of Spinlab, Inc. who also furnished the pulling mechanism, ion chamber and preamplifier, and electrometer used in the development work.

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Letters to the Editor

Stress-Relaxation Method of Determining the Accessibility of Fibers to Hydrochloric Acid

Department of Textile Industries
The University, Leeds 2, England
October 24, 1956

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In an interesting paper recently, Lemiszka and Whitwell [2] have reported an attempt to determine relative accessibilities of fibers to hydrochloric acid from measurements on stress relaxation in that reagent. Making the reasonable assumption that the HCl will preferentially hydrolyze bonds in the disordered regions, they argue that if the logarithm of the stress is plotted against time, a curve should result that approaches linearity when all the accessible stress-bearing bonds have been ruptured. If this portion of the curve is extrapolated to the ordinate, the intercept is proportional to the fraction of bonds initially present in the nonaccessible regions. The accessibilities of nylon and cellulosic fibers have been measured with this technique as a function of temperature and acid concentration. Although Lemiszka and Whitwell are careful to point out that their values are only relative ones and are dependent on the experimental conditions, there are several rather unsatisfactory features about this method that are worthy of comment.

First, the accessibility is being determined on samples that have been extended and allowed to relax in water for 24 hr. and are therefore not necessarily identical to the original samples; the influence on the accessibility of the preliminary extension and relaxation is unknown.

Again, "accessibility" is a vague term, but it is usually taken as being a quantitative measure of the maximum amount of material that is free to interact with a given reagent; i.e., it is a measure of an equilibrium quality of the fiber, and should not relate to a dynamic, or rate, quality. It is by no means certain that in the stress-relaxation method an equilibrium property is being measured. From the curves presented (especially Figure 2), only an approximation to a constant rate is ever attained. Accessibilities of nylon to HCl are apparently determined from extrapolation of lines determined over the time interval 1000–1500 min., whereas with cellulosic fibers the time interval 2000–5000 min. is used. The accessibility will obviously vary with the portion of the curve chosen as the "straight" line.

Additional evidence that their method does not measure the usual equilibrium accessibility comes from their results on nylons of varying degrees of cold-drawing. They find that the accessibility to 1 *N* HCl at 30° C. falls from 0.70 for undrawn nylon to 0.04 for a nylon of draw ratio 6.0. This enormous decrease in accessibility finds no support in measurements of the equilibrium uptake of HCl by nylon. Wall and Beresniewicz [5] find that 85–90% of the amino-end-groups of undrawn nylon 66 are accessible to HCl at pH 2, and although there are no data on absorption by the same sample in the drawn and undrawn states, almost 100% of the amino-groups of a typical drawn nylon 66 are also accessible to HCl [4]. There is thus no appreciable difference in the equilibrium accessibility to HCl at pH 2 between drawn and undrawn nylon 66, and it is most unlikely that an increase in the acid strength would cause a twentyfold *decrease* in the accessibility of the drawn sample.

At least three factors are involved in the stress relaxation, any one of which may affect the rate, and hence the apparent accessibility. The reaction must proceed by the following stages: (1) penetration of the fiber by the acid, (2) hydrolysis of the polymer, (3) relaxation and redistribution of strain following the alteration in the number of bonds bearing the stress.

Each of these processes may be complicated in various ways that have been ignored. The penetration of

the fiber is tacitly assumed to be uniform and complete, but this is not inevitable. Topochemical phenomena, such as the skin effects of regenerated cellulose fibers or the surface hydrolysis of polyethylene terephthalate fibers, may occur.

The rate of hydrolysis will also affect the apparent accessibility, as may be deduced from the data on the relative accessibilities of nylon and ramie fibers to acids of different concentrations. Ramie fibers are classed by the stress-relaxation method as being completely inaccessible to HCl over the concentration range $10^{-4} - 10^{-1}$ N; the accessibility is measurable only with 0.5 N HCl. Similarly, although undrawn nylon was apparently hydrolyzed by 1 N HCl, if the relaxation had been performed with 10^{-2} N HCl, which does not hydrolyze nylon fibers [4], nylon would have been classed as 100%, instead of 30%, inaccessible. The apparent accessibilities to different concentrations of reagent may be related to the distribution of lateral order, as suggested by Howsmon and Sisson [1], but this cannot be taken for granted. What is being measured is not so much physical accessibility as susceptibility to chemical attack, and a distinction between the two would be useful. This criticism also applies to many other chemical methods of measuring accessibilities, of course.

The third factor that may influence the method, the redistribution of strain that may follow rupture of the primary bonds, has also been ignored. The tacit assumption that the stress will depend directly on the number of main-chain bonds broken may be invalidated by redistribution of the secondary valence forces.

The stress-relaxation method is apparently capable of ranking cellulosic fibers in the same order of accessibility as other methods, but the ambiguities and uncertainties inherent in the interpretation of the results make it of doubtful value, without further confirmation, as a means of measuring quantitative accessibilities. For this purpose, moisture sorption, which has frequently been advocated in the past and has recently been shown to provide an accurate measure of the absolute crystallinity of cellulose [3], seems to have no equal for convenience and accuracy.

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LESLIE VALENTINE

Reply to Valentine Letter

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December 18, 1956

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Thank you for the opportunity of reading the recent letter from Dr. L. Valentine regarding the paper by the undersigned published in TEXTILE RESEARCH JOURNAL [4]. The implication that there are shortcomings in any method for accessibility does not surprise or particularly alarm us. We carefully noted that the values reported were relative and dependent upon experimental conditions, as also noted by Howsmon (see subsequent remarks). An additional stress-relaxation program, intended to elucidate the short-time stress-relaxation process, is in progress; some of the following remarks will refer briefly to this work.

Dr. Valentine's detailed comments will be discussed in the order in which they appear in his letter.

There is no pretense that anything but an arbitrary, water-equilibrated, datum is involved. Since some conditional datum is required and since this one is easily reproduced, it does not seem inadequate. It also has the advantage, for textile rayon, of producing a swollen state unchanged by the addition of HCl in concentrations up to 5 N [2].

The relative accessibility values reported may, in a minor way, depend upon the portion of the curve chosen. However, there seems to be no need to

apologize for the extrapolation technique. It has been used by many eminent workers in this field; for example, see [3, 5, 6, 7].

The questioning of different treatment times for cellulose and nylon samples seems to imply that Dr. Valentine expects that the rates of stress relaxation of these two materials should be identical. It is hard to credit this conclusion on our part.

The work of Wall and Beresniewicz [8] is concerned with the total numbers of end-groups in undrawn nylon available to HCl over an arbitrary time period of 10 to 14 days. These authors do not give information regarding the HCl absorption with time. Thus they intend to measure accessibility when all crystalline portions which can be penetrated have been so affected. In fact they state the existence of "certain regions which are inaccessible to penetration by aqueous hydrochloric acid"; inaccessible in this case obviously refers to the state at the end of 10 to 14 days treatment, while we are concerned solely with the situation at the beginning of treatment. Thus Dr. Valentine's remarks concerning end-groups do not seem pertinent to any method for relative accessibility in the sense in which we have defined it [4], p. 948.

Topochemical effects are indeed interesting and important. However, preliminary results, not yet published, indicate that "skin effects" do not interfere with the stress-relaxation picture. The relative quantity of amorphous and crystalline regions *seems* to react as if unaware of the existence of a skin. Confirmation of this tentative conclusion must await further theoretical work, under current development by R. E. Heitman and one of the undersigned.

Current work would indicate that the stress relaxation of nylon in HCl should not be termed hydrolysis, but the rate of the process certainly does depend upon the pH of the reagent. A normality of 10^{-2} does not rapidly affect drawn nylon, which is the reason for our use of a higher concentration. Dr. Valentine's statement "although undrawn nylon was apparently hydrolyzed by 1 *N* HCl, if the relaxation had been performed with 10^{-2} *N* HCl, which does not hydrolyze nylon . . . " is of course a contradiction. The situation where one concentration produces reaction but a lower one produces *none* is of course impossible; the rate can diminish, but not to zero.

In contrast to the views expressed by Dr. Valentine, we would be inclined to conclude that, for nylon in *N* HCl, physical accessibility is exactly what is being measured. Experiments in these laboratories have shown that a reversible reaction is involved. This could hardly be true if chemical hydrolysis had occurred with the fibers under stress.

It is interesting to note that Dr. Valentine has "information (whereby) moisture sorption, which has frequently been advocated in the past and has recently been shown to provide an accurate measure of the absolute crystallinity of cellulose, seems to have no equal for convenience and accuracy." P. H. Hermans has stated that the relation between moisture sorption and amount of amorphous material is a first approximation [1]. Howsmon [2] states that "since all the determinations of cellulose accessibility are relative, no attempt has been made to place the method on an absolute scale. . . . The best modification . . . seems to lie in the concept of 'accessibility';" he goes on to define accessibility as we did. Thus this suggestion of the establishment of a true standard is extremely interesting, particularly since it is so far more difficult to accomplish an accurate measure rather than to effect a mere improvement of precision.

We trust that these remarks may clarify our position, to the extent of our present knowledge, on the questions posed by Dr. Valentine.

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